

(SERIAL)

# RESEARCH IN PROGRESS

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## U.S. ARMY RESEARCH OFFICE

CHEMISTRY, BIOLOGICAL SCIENCES, ENGINEERING SCIENCES,  
METALLURGY AND MATERIALS SCIENCE,  
EUROPEAN RESEARCH PROGRAM



Department of the Army  
U.S. Army Materiel Development and Readiness Command

# **RESEARCH IN PROGRESS**

**BETWEEN 1 APRIL 1980 AND 31 MARCH 1981**

## **U.S. ARMY RESEARCH OFFICE**

**(REPORT EXEMPT FROM REPORTS CONTROL  
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## **FOREWORD**

The U.S. Army Research Office, under the U.S. Army Materiel Development and Readiness Command (DARCOM), is responsible for coordinating and supporting research in the physical and engineering sciences, in materials science, geosciences, biology, and mathematics.

This report describes research directly supported by the Army Research Office as well as research supported through this office by the Defense Advanced Research Projects Agency, several DARCOM and other Army commands, and the Defense Nuclear Agency.

The present volume includes the research program in metallurgy and materials science, mechanics and aeronautics, chemistry and biological sciences. It covers the 12-month period from 1 April 1980 to 31 March 1981. This issue will be the last to cover the above disciplines in a separate volume. The combined volume, covering also physics, electronics, mathematics and geosciences will be published as of 30 June 1982, and subsequent volumes will be published annually.

Additional copies of this report may be requested by writing to this office, attention DRXRO-IP.

DUFF G. MANGES  
Colonel, OD  
Commanding

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*IV*

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# LIST OF ABBREVIATIONS

|          |                                                                     |
|----------|---------------------------------------------------------------------|
| AFLRL    | Army Fuels and Lubricants Research Laboratory                       |
| AFOSR    | Air Force Office of Scientific Research                             |
| AMES     | Ames Aeromechanics Laboratory, AVRADCOM                             |
| AMMRC    | Army Materials and Mechanics Research Center                        |
| AMRDC    | Army Medical Research and Development Command                       |
| AMRDL    | Army Air Mobility Research and Development Laboratories             |
| APG      | Aberdeen Proving Ground                                             |
| ARI      | Army Research Institute                                             |
| ARL      | Army Aeromedical Research Laboratory                                |
| ARRADCOM | Army Armament Research and Development Command-Dover                |
| ASL      | Atmospheric Sciences Laboratory, ERADCOM                            |
| AVRADCOM | Aviation Research and Development Command                           |
| BMDATC   | Ballistic Missile Defense Advanced Technology Center                |
| BML      | Army Medical Bioengineering Research and Development Laboratory     |
| BRL      | Ballistic Research Laboratories, ARRADCOM                           |
| BWL      | Benet Weapons Laboratories, ARRADCOM                                |
| CENCOMS  | Center for Communications, CORADCOM                                 |
| CERL     | Construction Engineering Research Laboratory                        |
| CORADCOM | Communications Research and Development Command                     |
| CRREL    | Cold Regions Research and Development Command                       |
| CSL      | Chemical Systems Laboratory, ARRADCOM                               |
| CSTA     | Combat Survey and Target Acquisition Laboratory, ERADCOM            |
| DARCOM   | Army Materiel Development and Readiness Command                     |
| DOE      | Department of Energy                                                |
| DPG      | Dugway Proving Ground                                               |
| EOARD    | European Office of Aerospace Research and Development               |
| ERADCOM  | Electronics Research and Development Command                        |
| ETDL     | Electronics Technical and Devices Laboratory, ERADCOM               |
| EUSTIS   | Eustis Applied Technology Laboratory, AVRADCOM                      |
| FCSCWSL  | Fire Control and Small Caliber Weapons Systems Laboratory, ARRADCOM |
| FJSRL    | Frank J. Sellers Research Laboratory                                |
| HDL      | Harry Diamond Laboratories                                          |
| HEL      | Human Engineering Laboratories                                      |
| LAIR     | Letterman Army Institute of Research                                |
| LANGLEY  | Langley Structures Laboratory, AVRADCOM                             |
| LCWSL    | Large Caliber Weapons Systems Laboratory                            |
| LEWIS    | Lewis Propulsion Laboratory, AVRADCOM                               |
| MERADCOM | Army Mobility Equipment Research and Development Command            |
| MICOM    | Army Missile Research and Development Command                       |
| NLABS    | Natick Research and Development Center                              |
| NSWC     | Naval Surface Weapons Center                                        |
| NVEOL    | Night Vision and Electro-Optics Laboratory                          |
| NWC      | Naval Weapons Center                                                |
| ONR      | Office of Naval Research                                            |
| SC       | Scientific Cognizance                                               |
| SL       | Scientific Liaison                                                  |
| TARADCOM | Tank-Automotive Research and Development Command                    |
| TSC      | Technical Support Center                                            |
| USATHAMA | Army Toxic and Hazardous Materials Agency                           |
| WADC     | Wright Air Development Center                                       |
| WES      | Waterways Experiment Station                                        |
| WRAIR    | Walter Reed Army Institute of Research                              |
| WSMR     | White Sands Missile Range                                           |
| WVLT     | Watervliet Arsenal                                                  |

# I CHEMISTRY

## A. INORGANIC CHEMISTRY, ANALYTICAL CHEMISTRY, AND ELECTROCHEMISTRY

### 15611 STUDIES ON THE SYNTHESIS OF BORON HYDRIDE

S. G. Shore  
Ohio State University  
SC: MICOM, BRI

A procedure has been developed for converting  $B_2H_6$  to  $B_{10}H_{14}$ . It can be carried out in a single reactor. This procedure is developed to the point where one can consistently obtain  $B_{10}H_{14}$  in 45% yields of the crude product. Refined  $B_{10}H_{14}$  is obtained in 43-40% yields by recrystallization from hexane.

#### Reports:

- No. 1 in previous edition.
2. A Neutron Diffraction Study of Tris (methylphenylphosphine) [tetrahydroborato(1-)] Copper,  $Cu-P(C_6H_5)_3CH_3(BH_4)$ : The First Accurate Characterization of an Unsupported Metal-Hydrogen-Boron Bridge Bond, by Fusao Takusagawa et al.
3. Hexaborane (10) Derivatives: Relative Acidities of  $2-CH_3B_6H_{10}$  and  $2-BrB_6H_{10}$ ; NMR Spectra of  $2-CH_3B_6H_{10}$ ,  $2-BrB_6H_{10}$ , and  $(THF)_2Mg(2-CH_3B_6H_{10})_2$ , by Randall J. Remmel et al, *Inorg Chem* 20, 1270 (1981).
4. New, Systematic, Good Yield Syntheses of Boron Hydrides: Preparation of  $B_2H_6$  and  $B_4H_{10}$ . A Practical Conversion of  $B_2H_6$  to  $B_{10}H_{14}$ , by John B. Leach et al, *J Am Chem Soc* 103, 988 (1981). AD A097 907
5. Studies on the Conversion of Pentaborane(9) to Decaborane(14), Part II, by Sheldon G. Shore, 5p, TR. AD A089 957
6. Studies on the Conversion of Pentaborane(9) to Decaborane(14), Part III, by Sheldon G. Shore et al, Dec 80, 5p, figures, TR. AD A092 995
7. New, Systematic, Good Yield Syntheses of Boron Hydrides: Preparation of  $B_2H_6$  and  $B_4H_{10}$ . A Practical Conversion of  $B_2H_6$  to  $B_{10}H_{14}$ , by John B. Leach et al, *J Am Chem Soc*.

### 15783 THE SYNTHESIS AND STRUCTURE OF POLYPHOSPHAZENES

Harry R. Allcock  
Pennsylvania State University  
SL: AMMRC SC: NLABS, MERADCOM, HDL, ARRADCOM

Efforts have continued on exploring synthesis routes for the preparation of phosphazene high polymers with metallic units incorporated into the side group structure. The long-range objective is the development of polymer systems that conduct electricity or which can function as catalyst systems in solution or in the solid state. Two specific approaches are being pursued: (1) the attachment of metal atoms to organic spacer units that are linked to the skeletal phosphorus atoms, and (2) the linkage of metal atoms directly to the skeleton through phosphorus-metal bonds. A major synthetic development has been the synthesis of cyclophosphazenes with iron atoms bonded directly to the phosphorus atoms of the ring. This is seen as a prototype reaction for the preparation of high polymers with "outrigger" chains of metal atoms flanking a phosphazene skeleton. Similar model compounds have now been prepared with ruthenium or rhenium atoms attached to the ring, and a species with both iron and ruthenium linked together as side group atoms has been isolated. These syntheses constitute a general reaction with widespread possibilities for new polymer synthesis.

#### Reports:

- No. 1-5 in previous editions.
6. Polymerization of Cyclic Phosphazenes and Other Inorganic Ring Systems, by Harry R. Allcock, *Polymer*.
7. Synthesis and Molecular Structure of Two Cyclotriphosphazenes with Phosphorus-Iron Bonds, by Harry R. Allcock et al, *Inorg Chem*.
8. Polyphosphazene Elastomers, by H. R. Allcock.
9. Controlled Synthesis of Organic-Inorganic Polymers that Possess a Backbone of Phosphorus and Nitrogen Atoms, by Harry R. Allcock, *Makromol Chem*.



**15976 ELECTROGENERATED CHEMILUMINESCENCE**

A. J. Bard

University of Texas at Austin

SL: FCSCWSL SC: LCWSL, ETDL, CSL

Further research has been conducted in the area of polymer-coated electrodes. In particular, investigations have been made of species and the use of ecl data, in addition to the electrochemical results, for elucidating the mechanism of reactions occurring at modified electrodes. Nafion-coated electrodes (pyrolytic graphite (PG), glassy carbon (GC) or Pt substrates) were used. ECL from surface-confined  $\text{Ru}(\text{bpy})_3^{2+}$  is observed when a Pt/Nafion,  $\text{Ru}(\text{bpy})_3^{2+}$  electrode (or similarly modified GC or Pt) is scanned to positive potentials in an aqueous solution containing oxalate ions. This ecl emission exhibits a very unusual behavior which is different than the ecl from the same species in solution. For instance, when the modified electrode potential is stepped positively, a light intensity peak is observed and the ecl then reaches a steady state level. When the potential is stepped back to more negative values (or the circuit is opened), a large increase in light intensity is observed before it eventually drops to zero. On the basis of the combined electrochemical and ecl data, a model has been developed which accounts for all of the experimental observations.

**Reports:**

No. 1-2 in previous edition.

3. Electrogenated Chemiluminescence. 36. The Production of Steady Direct Current ECL in Thin Layer and Flow Cells, by George H. Brilmyer et al, *J Electrochem Soc* 127, 104 (1980). AD A090 859
4. Electrochemical and Photoelectrochemical Studies of Excess Electrons in Liquid Ammonia, by Allen J. Bard et al, *J Phys Chem* 84, 1262 (1980). AD A090 858
5. Application of Photothermal Spectroscopy to in-Situ Studies of Films on Metals and Electrodes, by George H. Brilmyer et al, *Anal Chem* 52, 685 (1980). AD A090 860.
6. Simultaneous Determination of Quantum Efficiency and Energy Efficiency of Semiconductor Photoelectrochemical Cells by Photothermal Spectroscopy, by Akira Fujishima et al, *J Electrochem Soc* 127, 840 (1980). AD A090 861
7. Successive Electron Transfers to Molecules with Identical Interacting Centers-The Electroreduction of  $\alpha$ ,  $\omega$ -9, 9'-Dianthrylalkanes, by Kingo Itaya et al, *Zeitschrift für Physikalische Chemie Neue Folge* 112, 1 (1978). AD A090 862

**16116 STUDIES OF THE LITHIUM ELECTRODE IN OXYCHLORIDE SOLVENTS**

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EIC Corporation

SL: ETDL SC: MERADCOM, HDL

Film growth kinetics and some properties were studied on Li electrodes in  $\text{SOCl}_2$  solvent containing: (a) 1.8M  $\text{LiAlCl}_4$ ; (b) 1M  $\text{LiAlSCl}_2$ ; (c) a + 0.1M  $\text{H}_2\text{O}$ . The influence of some additives was also studied. Two types of electrode with different cell configuration were examined: (1) Foil electrodes pressed on Ni grid in resemblance to those normally used in Li batteries. Cylindrical symmetry electrode configuration was used. (2) Two 0.5" Li rods facing each other and exposing one surface to solution. A fresh surface on one electrode was created by scraping with a Ni peg. Experiments on fresh surface Li electrodes were carried out using a newly constructed all-glass cell and cutting device which minimize metallic contamination of the 1.8M  $\text{LiAlCl}_4/\text{SOCl}_2$  solutions used. The accuracy and precision of the capacitance measurements were improved by using high frequency (1 MHz) rectangular alternating current. Reproducible film properties were found during the initial two days of the growth period with a parabolic growth rate. The kinetics of film growth were found to be dependent on electrolyte storage in the glove box.

**Reports:**

1. Properties of Solid Electrolyte Films Grown on Fresh Surface Lithium Electrodes, by M. Yaniv et al.

**16191 NEW SYNTHETIC TECHNIQUES IN BORON CHEMISTRY**

Larry G. Sneddon

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SC: MERADCOM

Efforts are continuing to exploit the discovery that certain transition metal complexes can catalyze the reactions of boron hydrides and alkynes. Two different complexes have now been studied in some detail,  $\text{Ir}(\text{CO})\text{Cl}(\text{PPh}_3)_2$  and  $(\text{R}_2\text{C}_2)\text{Co}_2(\text{CO})_6$ , and both have been found to catalyze the reaction of pentaborane(9) and alkynes. These results are especially important in light of the work which has shown that these alkenylpentaboranes can be



converted in high yields to mono-carbon carboranes. Both the metal catalyzed syntheses and the carborane conversion reactions are discussed in full in a recently submitted paper. Results similar to those described above have been obtained with a variety of catalysts, and in addition it was found that several of these catalysts will activate the reactions of alkynes and carboranes, as well as boranes, to yield the corresponding B-alkenyl derivatives. The metal catalyzed reactions represent the first practical synthetic route to B-alkenylcarboranes. In addition, it has been shown that the degree of substitution of the carborane can be controlled by varying the reaction conditions and products have been obtained ranging from single substitution at only one boron site to complete substitution at all boron positions in the molecule. These carborane reactions will be described in a forthcoming paper.

**Reports:**

- No. 1 in previous edition.
- 2. Metal Atom Syntheses of Metalla-boron Clusters II: Synthesis of Cobaltathiaaborane and Cobaltadithiaaborane Complexes. Crystal and Molecular Structures of 2,3,6-( $\eta$ -C<sub>3</sub>H<sub>3</sub>):Co:SB<sub>3</sub>H<sub>3</sub> and 7,6,8-( $\eta$ -C<sub>3</sub>H<sub>3</sub>):CoS<sub>2</sub>B<sub>3</sub>H<sub>3</sub>, by George J. Zimmerman et al, *J Am Chem Soc*.
- 3. Transition Metal Catalyzed Reactions of Alkynes and Boron Hydrides: Synthesis of 2- (cis-2-Butenyl) pentaborane(9) and Its Conversion into Monocarbon Carboranes, by Robert Wilczynski et al, *J Am Chem Soc* 102, 2857 (1980). AD A092 693
- 4. Transition Metal Promoted Reactions of Boron Hydrides II. Synthesis and Thermolysis Reactions of Alkenyl-pentaboranes: A New Synthesis of Monocarbon Carboranes, by Robert Wilczynski et al.

## 16192 TWO-DIMENSIONAL MASS SPECTROMETRIC SEPARATION AND IDENTIFICATION OF COMPLEX MIXTURES

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SI: NLABS, AMMRC SC: CSI

The tandem double-focusing mass spectrometer is now complete with the second magnet installed and the computer-controlled linked-scanning circuit made operational. The replacement 23 kilogauss, 60b cm magnet for MS-I has been received, and installed. Addition of the second magnetic sector to complete the tandem double-focusing MS/MS instrument appears to have given a dramatic improvement in sensitivity by increasing

the signal/noise by one or two orders of magnitude. The collisional activation mass spectrum measured by MS-II has a broad distribution of ion kinetic energies because of the energy release in dissociation after CA; mass analysis using only the energy analyzer thus gives relatively broad peaks, with less than unit mass resolution even below mass 100. The addition of the magnetic analyzer makes MS-II double-focusing, so that ions of the same mass but different energies are focused together. Mass spectrometers coupled in series (MS/MS) offer a promising technique for quantitative analysis of trace constituents in complex mixtures. Despite the relatively high cost and complexity of MS/MS instruments, this technique appears to be particularly promising in terms of the sensitivity, specificity, and speed of analysis for targeted compounds in body fluids, foods, and other biological mixtures. Fragmentations of even-electron ions of lifetimes  $\leq 10^{-5}$  s caused by electron and chemical ionization and by collisional activation have been correlated. Proposed reaction classifications include: cleavage of one bond with charge migration; cleavage of one bond through cyclization-displacement; cleavage of two bonds in a cyclic ion with charge retention; and cleavage of two bonds in an acyclic ion with rearrangement and charge retention. Such reactions are compared with those of odd-electron ions; despite a higher tendency for rearrangement, the decompositions of a positive even-electron ion, in particular as displayed in its collisional activation spectrum, have substantial utility for characterizing its structure.

**Reports:**

- No. 1-5 in previous editions.
- 6. Mass Spectrometry as a Separation Technique; The Analysis of Involatile Samples, by Fred W. McLafferty et al, *J Chromatography*.
- 7. Tandem Mass Spectrometry (MS/MS) in Trace Toxicant Analysis, by Fred W. McLafferty.
- 8. Unimolecular Decompositions of Even-Electron Ions, by F. W. McLafferty, *Org Mass Spectrum* 15, 114 (1980).

## 16256 THE PREPARATION AND PROPERTIES OF LIGAND-BRIDGED METAL COMPLEXES

Thomas J. Meyer  
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SI: CSI SC: NLABS

A series of metallopolymers have been prepared by the reaction between poly - (4-vinylpyridine)

(mw ~ 3900) and  $[\text{Ru}(\text{trpy})(\text{bpy})(\text{OH}_2)]^{2+}$  (trpy is 2, 2', 2''-terpyridine and bpy is 2, 2' bipyridine). The synthetic procedure allows for the degree of metallation of the polymer to be controlled in a systematic and reproducible way. The degree of metallation in a series of samples was determined by  $^1\text{H}$  NMR, uv-vis spectroscopy, elemental analysis and ESCA. The redox properties of the materials as thin films on electrode surfaces were investigated electrochemically. The results of uv-visible absorption and emission studies in solution suggest that in samples where the degree of metallation is low, the optical properties of the polymer-bound sites are essentially those of the related monomer,  $\text{Ru}(\text{trpy})(\text{bpy})\text{py}^{2+}$ , but at high degrees of metallation, evidence for interactions between sites has been obtained. Laser flash photolysis on a series of unsymmetrical ruthenium dimers has provided evidence for directed, intramolecular excitation energy transfer by a one-electron pathway for mixed-valence,  $\text{Ru}^{\text{II}}\text{-Ru}^{\text{III}}$  dimers and by simple energy transfer for  $\text{Ru}^{\text{II}}\text{-Ru}^{\text{II}}$  dimers. The effects of solvent on the energies of charge transfer bands can be treated using electron transfer theory. Experimental tests of the theory have been made using mixed-valence dimers, mixed metal ion pairs, organic donor-acceptor complexes, and metal-ligand or ligand-metal charge transfer transitions.

#### Reports:

No. 1-2 in previous edition.

3. Solvent Effects on Charge Transfer Transitions in Mixed Valence, Organic Donor-Acceptor, and Related Chemical systems, by B. Patrick Sullivan et al, *Nouveau J De Chimie* 4, 643 (1980). AD A095 219
4. Selectivity and Directed Charge Transfer Through an Electroactive Metallopolymer Film, by Charles D. Ellis et al, *J Am Chem Soc.*

### 16266 STUDIES OF SILICON-NITROGEN-PHOSPHORUS COMPOUNDS INCLUDING A NEW SYNTHESIS OF PHOSPHAZENES

Robert H. Neilson  
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SL: AMMRC SC: MERADCOM, NLABS, CSI

The principal area of investigation has focused on the new synthesis of poly(dialkylphosphazenes) from P-trifluoroethoxy-N-trimethylsilylphosphinimines via the elimination of  $\text{Me}_3\text{SiOCH}_2\text{CF}_3$ .

The new polymer, poly(dimethylphosphazene)  $(\text{Me}_2\text{PN})_n$ , has been fully characterized. Various reaction conditions for polymer formation, including thermolysis of the phosphinimine (where  $\text{R}=\text{R}'=\text{Me}$ ) in glass ampules, in a stainless steel bomb, under a neat reflux, and in refluxing benzene have been investigated. A prime objective has been finding the best method for the large scale synthesis of this novel new polymer. The scope of the new polyphosphazene synthesis has been extended to include the preparation of polyphosphazenes with mixed alkyl and/or aryl as the symmetrically substituted polymers reported previously. The conditions required for the thermolysis of the respective N-silyl-phosphinimines are more severe than those reported for the thermolysis of  $\text{Me}_2\text{P}(\text{OCH}_2\text{CF}_3) = \text{NSiMe}_3$ .

#### Reports:

No. 1-6 in previous editions.

### 16631 IR PHOTOACOUSTIC STUDIES OF SURFACE REACTIONS

Manfred J. D. Low  
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SL: LCWSL, CSL SC: MERADCOM

This project has two major thrusts. The first is to study the chemistry of benzotriazole as corrosion inhibitor of copper. The second and major thrust is to develop the technique of infrared photoacoustic spectroscopy (IR/PAS) to study the surfaces of samples that are rough and opaque. A dispersive infrared photoacoustic spectrometer system used to record infrared spectra over the 4000-700  $\text{cm}^{-1}$  range, and a Fourier transform infrared photoacoustic spectrometer system were constructed. After the Fourier transform photoacoustic spectrometer was made operational it was found that it and the new detection system would not function properly because of excessive low frequency vibrations of the environment. It was necessary to remove the vibration isolation system from the dispersion spectrometer system, because no other isolation system was available, and to transfer it to the Fourier transform system. Both spectrometers had to be partially disassembled. The dispersive system is now inoperable, but the Fourier transform spectrometer has been again completed. Work has also been almost completed on the new Schlieren detection system; it also had to

be rebuilt to make it stronger mechanically and less sensitive to vibration. Work has begun on the chemical aspects of the research. Compounds of benzotriazole and various copper compounds have been prepared, and some infrared Fourier transform spectra have been recorded.

#### Reports:

No. 1-6 in previous edition.

7. Dispersive Photoacoustic Spectroscopy of Solids in the Infrared Range, by M.J.D. Low et al, *J Photoacoustic Spectroscopy*.
8. A Detection System for Photoacoustic Spectroscopy, by M. J. D. Low et al, *Chem, Biomed, and Environ Instrumen*.

### 16865 A PHOTOACOUSTIC STUDY OF CHEMICALLY ACTIVE SYSTEMS

Barrie S. H. Royce  
Princeton University

SL: LCWSL SC: CSL, NIABS

The application of the new technique of Fourier transform photoacoustic spectroscopy to the study of condensed phase samples in the infrared spectral region between 4000 and 400  $\text{cm}^{-1}$  has been continued. The Fourier transform technique has made possible the spectral study of a wide range of materials that are hard to examine in other ways. Of particular interest at the present time are polymeric systems that are undergoing cure reactions. Two such systems that are being studied are RTV rubbers that contain fillers such as iron oxide or silica and acrylics that are used to coat optical fibers. The advantage of the photoacoustic technique in these studies is that the filled materials are strongly light scattering and have therefore proved impossible to examine using either conventional transmission or reflection FTIR methods. As the photoacoustic technique measures only the absorbed energy the light scattering problem is minimal with this method and high resolution spectra can be obtained. These measurements are to be extended to study the effect of the filler material on the polymerization process occurring in the RTV. This material is an important potting compound for microelectronic systems and this study should permit filler materials to be chosen so as to optimize the properties of the cured two phase material. PAS has also permitted the study of acrylic material that has been photocured onto optical fibers. As this system is again strongly

light scattering a spectroscopic study of the fiber coating by conventional means had not been possible and even Raman spectroscopy did not yield good spectral information. The success of the FTIR-PAS method will permit optical techniques to be employed for quality control of fiber coatings.

#### Reports:

No. 1 in previous edition.

2. Fourier Transform Infrared Photoacoustic Spectroscopy of Solids, by B.S.H. Royce et al, *1980 Ultrasonics Symp Proc*, p652, Nov 80, AD A097 432
3. Elimination of Dispersion-Induced Distortion in Infrared Absorption Spectra by Use of Photoacoustic Spectroscopy, by G. Laufer et al, *Appl Phys Let* 37, 517 (1980), AD A093 918
4. Quantitative Fourier Transform Infrared Photoacoustic Spectroscopy of Condensed Phases, by Y. C. Teng et al, *Opt Soc Am*.
5. Fourier Transform Infrared Photoacoustic Spectroscopy of Condensed Phases, by B. S. H. Royce et al.

### 17042 CHEMICAL IONIZATION MASS SPECTROMETRY: NEW METHODOLOGY

Donald F. Hunt  
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SL: CSI, MERADCOM, NIABS

To sequence neuropeptides at the picomole level an approach is being evaluated which utilizes both electron capture negative ion mass spectrometry and collision activated dissociation of dipeptide carboxylate anions. The procedure can be illustrated with the model peptide, Tyr-Gly-Gly-Phe-Met. The sample (100 pmole) is hydrolyzed in a melting point capillary with 5  $\mu\text{l}$  of 0.03 N HCl and the resulting mixture of peptides is converted to  $\text{d}_0/\text{d}_1$ -N-acetyl peptide O-pentafluorobenzyl esters. Under electron capture negative ion chemical ionization conditions the O-perfluoro benzyl esters capture thermal electrons and dissociate to dipeptide carboxylate anions. The sample ion current produced in the negative ion mode under these conditions exceeds that obtained in the positive ion mode by a factor of 100.

#### Reports:

1. Collision Activated Decompositions of Negative Ions in Mixture Analysis with a Triple Quadrupole Mass Spectrometer, by Donald F. Hunt et al, *Anal Chem* 52, 386 (1980), AD A092 497
2. Sequence Analysis of Oligopeptides by Secondary Ion Collision Activated Dissociation Mass Spectrometry.

**17069 REACTIONS OF COORDINATION COMPOUNDS IN THE SOLID STATE**

John C. Bailar, Jr.  
University of Illinois  
SC: CSL

The object of the project is to reveal the kinetics and mechanics of some solid state reactions, such as (1)  $[\text{Pt}(\text{Me}_2\text{S})_4][\text{PtCl}_4] \rightarrow 2[\text{Pt}(\text{Me}_2\text{S})_2\text{Cl}_2]$  and (2)  $[\text{Pt}(\text{NH}_3)_4][\text{PtCl}_4] \rightarrow 2[\text{Pt}(\text{NH}_3)_2\text{Cl}_2]$ . Several complexes have been prepared on the basis of an assumption of how the reactions may go. After several operations, complexes which seem to be the intermediates of reactions A and B have been isolated. At present, the intermediates are being characterized and their crystal structures are being determined by x-ray diffraction.

**17108 SYNTHETIC & MECHANISTIC STUDIES IN FLUORINE CHEMISTRY. NEW DEVELOPMENTS IN ORGANONITROGEN FLUORINE CHEMISTRY**

Darryl DesMarteau  
Kansas State University  
SL: AMMRC SC: CSL

The reaction of halogenated nitriles with a 1:1 mixture of  $\text{ClF}:\text{F}_2$  proceeds in high yield to form N-chloro-N-fluoroalkylamines of the type  $\text{RCF}_2\text{NCIF}$ . Characterization of the new compounds  $\text{CF}_3\text{CF}_2\text{NCIF}$ ,  $\text{CF}_3\text{CF}_2\text{CF}_2\text{NCIF}(\text{CF}_3\text{NCIF})_2$  and  $\text{CCl}_3\text{CF}_2\text{NCIF}$  has been completed along with an improved synthesis of  $\text{ClCF}_2\text{NCIF}$ . The chlorofluorination reaction proceeds mainly via the initial formation of  $\text{RNCI}_2$ , which is then fluorinated in a new type of reaction to give  $\text{RNCIF}$  and  $\text{ClF}$ . The reaction of elementary fluorine with ten perhalogenated compounds containing carbon-nitrogen double bonds has been studied. The reactions were carried out without added catalysts in a static system. With  $\text{CF}_3\text{N}=\text{CF}_2$ ,  $(\text{CF}_3)_2\text{NCF}=\text{NCF}_3$ ,  $\text{SF}_5\text{N}=\text{CF}_2$ ,  $\text{CF}_2=\text{NCl}$ ,  $\text{CF}_3\text{CF}=\text{NCl}$  and  $\text{CF}_2=\text{NF}$  a simple addition of fluorine to the carbon-nitrogen double bond was observed forming the respective N-fluoroamines in

high yield. Two imines,  $\text{CF}_3\text{CF}=\text{NF}$  and  $(\text{CF}=\text{NF})_2$ , were unreactive under the same conditions and reactions of  $\text{CF}_3\text{CF}_2\text{CF}=\text{NF}$  and  $(\text{CF}_3)_2\text{C}=\text{NF}$  were explosive. The reaction of N-chloro-N-fluoroalkylamines with mercury has been studied with  $\text{ClCF}_2\text{NCIF}$ ,  $\text{CF}_3\text{NCIF}$ ,  $\text{CF}_3\text{CF}_2\text{NCIF}$ ,  $\text{CF}_3\text{CF}_2\text{CF}_2\text{NCIF}$  and  $(\text{CF}_3\text{NCIF})_2$ . In the absence of solvents, all but  $\text{CF}_3\text{NCIF}$  undergo dehalogenation to form the corresponding N-fluoroamines in good yield.

**Reports:**

1. The Addition of Fluorine to Perhalogenated Compounds Containing Carbon-Nitrogen Double Bonds, by Akira Sekiya et al, *J Fluorine Chem* 17, 463 (1981).
2. The Reaction of N-Chloro-N-Fluoroperhaloalkylamines with Mercury. A Facile Synthesis of N-Fluoroamines and N-Fluoroamines, by Akira Sekiya et al, *J Org Chem* 46, 1277 (1981). AD A099 548
3. Perfluoromethanamine Ion, by Shi-Ching Chang et al, *J Am Chem Soc.*
4. Some Novel Oxime Ethers. The Synthesis of 1, 1-Difluoro-2-Alkoxymethanimines, by William Y. Lam et al, *J Fluorine Chem.*
5. Novel Sulfur-Nitrogen Fluorine Compounds. Synthesis and Properties of  $\text{SF}_5\text{NCIF}$ ,  $\text{SF}_5\text{NHF}$  and  $\text{FN}=\text{SF}_5$  and the Molecular Structure and Vibrational Analysis of  $\text{FN}=\text{SF}_5$ , by Darryl D. DesMarteau et al, *Inorg Chem.*

**17154 FUNDAMENTAL PROCESSES OCCURRING AT ELECTRODES**

Fred C. Anson  
California Institute of Technology  
SL: MERADCOM SC: CSL, ETDL, BWL

Electrochemical studies will be made with electrodes that have had their surfaces prepared by chemical modification to accept and bind particular molecules and complex ions. Emphasis will be placed on polymers and, especially, polyelectrolytes as surface binding agents. The attachment of multiply-charged reactants to electrode surfaces by electrostatic binding will be a major component of the research. The catalysis of electrode processes such as the reduction of oxygen by means of the reactants bound to electrode surfaces will be explored. The mechanism of electron transfer through polymeric matrices containing anchored redox active sites will be examined in electrode kinetic and photochemical experiments.



# BEST DOCUMENT AVAILABLE

A. Inorganic Chemistry, Analytical Chemistry, and Electrochemistry

1 Chemistry

## 17386 BASIC RESEARCH STUDIES IN NITROGEN-HALOGEN CHEMISTRY

Karl O. Christe  
Rockwell International  
SL: MICOM SC: CSL, LCWSL, AMMRC

A basic research program will be conducted in nitrogen-halogen chemistry. Areas to be studied include: (1) Synthesis and characterization of novel  $\text{NF}_4^+$  salts of high active fluorine content containing doubly and triply charged anions. (2) Investigation of new synthetic methods and solvents for these materials. (3) Synthesis and characterization of unique energetic  $\text{NF}_4^+$  salts as ingredients for high detonation pressure explosives. (4) Determination of the structural, spectroscopic, and thermodynamic properties of some of the compounds. (5) Synthesis and characterization of  $\text{N}_3\text{F}_4^+$ ,  $\text{NCl}_4^+$ , and  $\text{N}_3\text{F}_2^+$  salts and of novel  $-\text{NF}_2$  substituted molecules and ions. (6) Study of the reaction chemistry of  $\text{NF}_4^+$  salts and their conversion to novel hypofluorites. Studies are being made on energetic hypofluorites. The vibrational spectra of fluorine perchlorate,  $\text{FOClO}_4$ , were analyzed, a normal coordinate analysis was carried out, and thermodynamic properties were computed. The results are presently being summarized in manuscript form. The ionic hypofluorite,  $\text{CS}^+\text{FOSO}_3^-$ , was prepared by a recently reported method. If stable in anhydrous HF, it will be used for a metathetical reaction in an attempt to combine it with the energetic  $\text{NF}_4^+$  cation. Characterization of the covalent trifluoromethylazide,  $\text{CF}_3\text{N}_3$ , was completed and the results were written up as a manuscript. In the area of heptavalent iodine compounds, the reaction of  $\text{CsIO}_4$  with  $\text{ClF}_5$  has been studied. Whereas after short reaction times,  $\text{trans-CsIF}_4\text{O}_2$  was formed in low yield, longer reaction times produced mixtures containing mainly  $\text{CsIF}_6$ ,  $\text{CsIF}_5$  and  $\text{CsIO}_4$  and smaller amounts of  $\text{CsIF}_4\text{O}$  and cis and trans  $\text{CsIF}_4\text{O}_2$ .

### Reports:

1. Vibrational Spectra of  $^{19}\text{NF}_4\text{AsF}_6$  and General Valence Force Field of  $\text{NF}_4^+$ , by Karl O. Christe, *Spectrochim Acta* 36A, 921 (1980). AD A092 671
2. Simplified Synthesis of  $\text{NF}_4^+\text{SbF}_6^-$  by William W. Wilson et al, *J fluorine Chem* 15, 83 (1980).
3. Synthesis and Properties of  $\text{NF}_4^+\text{ClO}_4^-$  and  $\text{NF}_4^+\text{HF}^- \cdot n\text{HF}$  and Some Reaction Chemistry of  $\text{NF}_4^+$  Salts, by Karl O. Christe et al, *Inorg Chem* 19, 1494 (1980).

4. Inorganic Halogen Oxidizer Research, by K. O. Christe et al, Mar 81, 176p, figures, Annual TR: 1 Mar 80 - 28 Feb 81. AD A098 705

## 17398 SYNTHESIS OF POLYHEDRANES AND POLYHEDRAL CARBORANES

R. Bruce King  
University of Georgia  
SL: LCWSL SC: MICOM, BRL

The objective of the research is to develop new synthetic materials for the synthesis of polyhedral carboranes and polyhedranes. The following areas of chemistry will be investigated: (1) thermal and photochemical reactions of borane metal carbonyls with alkynes; (2) photochemical reactions of alkyne metal complexes with boranes; (3) preparation of metal complexes containing two or three  $\text{B}_3\text{H}_8$  ligands; (4) reactions of metal vapors with  $\text{BH}_3\cdot\text{L}$  and  $\text{B}_3\text{H}_7\cdot\text{L}$  derivatives; (5) photolysis of  $\text{Li}_2\text{C}_2$ ; and (6) the reactions of transition metal complexes with cyclobutadiene and triquinocene ligands. The major effort has been directed towards the syntheses of tetrahedrane derivatives  $[(\text{CH}_3)_3\text{E}]_4\text{C}_4$  ( $\text{E}=\text{Si}$  or  $\text{Sn}$ ). All of the synthetic approaches investigated used the reaction scheme based on the reagent  $\text{Li}_4\text{C}_4$  recently reported by Schleyer and co-workers and claimed to be tetralithiotetrahedrane. The general impression from these experiments is that  $\text{Li}_4\text{C}_4$  is too insoluble in appropriate solvents for reaction with halides to take place. All of the experiments gave a large amount of white solid which reacted violently with water and thus appeared to be unreacted  $\text{Li}_2\text{C}_2$  or  $\text{Li}_4\text{C}_4$ . These negative results suggest that further studies on the reactions of  $\text{Li}_4\text{C}_4$  or  $(\text{BrMg})_4\text{C}_4$  with  $(\text{CH}_3)_3\text{ECI}$  ( $\text{E}=\text{Si}$  and  $\text{Sn}$ ) to give tetrahedrane derivatives are not likely to be productive at the present time. Plans are being made to explore possibilities for the preparation of dialkylamino-substituted tetrahedranes  $(\text{R}_2\text{N})_4\text{C}_4$  through the photolyses of bis(dialkylamino)acetylenes.

## 17456 A NEW APPROACH TO HYDROGEN STORAGE: MOLECULAR POLYHYDRIDES

Robert H. Crabtree  
Yale University  
SL: MERADCOM SC: FIDL

A new approach to the storage of hydrogen will be investigated. Metal complexes of the type  $\text{LMH}_x$

(L=anchoring ligand; M=Mo or W) are to be anchored on to a polymer support, in the presence of a labile ligand  $L^1$  which can reversibly displace  $H_2$ . Basic studies on the nature of the M-H bond and the redox character of  $H_2$  addition are proposed to identify suitable systems and ligands. The synthesis of molecular polyhydrides has been examined with a view to their application in hydrogen storage and release. Initial investigations reveal that new hydrides of the type  $MoH_6(PR_3)_3$  (R=Cyclohexyl, *tert*-butyl) can be prepared from  $Li(Et_3BH)$ ,  $MoCl_4(thf)_2$  (thf=tetrahydrofuran), and  $PR_3$  only at -100 degrees. This method is also applicable to the known hydrides  $MH_4L_4$  (M=Mo or W; L=PMePh<sub>2</sub>, or 1/2 (Ph<sub>2</sub>PCH<sub>2</sub>)<sub>2</sub>). In these cases the yields have been increased from literature values ten fold (W) or three-fold (Mo) to 35 - 67%. Initial studies in this area suggest that one difficulty is the tendency of these compounds to dimerise on release of hydrogen.

#### 17650 STUDIES IN BORON CHEMISTRY

Bernard F. Spielvogel  
Andrew T. McPhail  
Duke University  
SC: BRL

The objective of the research is to fully characterize the chemical and physical properties of amine-carboxyboranes (boron analogs of amino acids). The hydrolytic, thermal and acid-base behavior of amine-carboxyboranes will be determined. The chemical reactivity of amine-carboxyboranes to form polyamides, complexes with transition metals, and as selective reducing agents will be explored. Measurements of pKa on  $H_2NBH_2CO_2H$ , the boron analog of glycine, and the series of N-methyl derivatives have been carried out in collaboration with Professor Robert Martin, University of Virginia. The series behave as very weak acids, pK's in the range of 8. In solution they form strong complexes with metal ions such as Cu. No evidence could be obtained for complexation via the nitrogen and is therefore in contrast to the behavior found for  $\alpha$ -amino acids. A complex  $ZnCl_2(Me_2NBH_2CN)_2$  has been synthesized from anhydrous  $ZnCl_2$  and  $Me_2NBH_2CN$  in ethanol. It has been characterized by x-ray crystallographic analysis. The complex rapidly undergoes

hydrolysis but no evidence for the hydrolysis of the CN to amide or acid was obtained.

#### Reports:

1. Basicity and Metal Ion Binding Capability of Amine-Carboxyboranes,  $R_2N^+BH_2COOH$ , Boron Analogs of Glycine and N-Methylated Glycines, by Kurt H. Scheller et al, *Inorg Chimica Acta*.
2. Antihyperlipidemic Activity of Amine Cyanoboranes, Amine Carboxyboranes, and Related Compounds, by I.H. Hall et al, *J Pharmaceutical Sci* 70, 339 (1981).

#### 17654 A STUDY OF GAS SOLID REACTIONS AND AIR POLLUTION DETECTORS

George G. Guilbault  
University of New Orleans  
SC: CSL, MERADCOM, LCWSL

An investigation will be made to find specific adsorbents for inorganic and organic chemicals and pollutants. The basic reaction of the chemical gaseous adsorbents with these substances will be studied using piezoelectric quartz oscillating crystals. The gas-solid reactions occurring at the sensor surface will be studied to determine the nature of the interaction (physical or chemical reaction, reversible or irreversible, etc.) and the selectivity of the interaction for the pollutant of interest.

#### 17715 ELECTROCHEMISTRY OF POLYMER FILMS. APPLICATIONS TO CHEMICAL ANALYSIS & ENERGY CONVERSION

James Q. Chambers  
University of Tennessee  
SL: MERADCOM SC: CSL

An investigation will be made of the electrochemistry of polymer films which can incorporate both electronic and ionic charge in a quasi-reversible fashion. Polymers with both electron donor and acceptor substituents will be studied and the characteristics of these materials which lead to electroactive polymer film electrodes determined.

**17745 LASER DETECTION OF CHEMICAL AGENTS**

Richard N. Zare  
Stanford University  
SL: BRL, CSL SC: CSL, NLABS

A fundamental study will be initiated of laser multiphoton ionization mass spectrometry as a new analytical tool. The compound of interest will be introduced into the source of a mass spectrometer where it is exposed to the intense radiation field of a tunable pulsed laser light source. At laser wavelengths corresponding to submultiples of real intermediate states, the molecule will absorb more than one photon, leading to ionization and fragmentation. The mass-to-charge ratio of the ions will be measured by the mass spectrometer. Considerable effort has been directed toward improving the multiphoton ionization time-of-flight (MPITOF) mass spectrometer. The sample inlet system was modified to minimize contamination from previously run samples and to allow a rapid change of samples. The flight tube of the mass spectrometer was lengthened to increase the resolution. The data-taking electronics are also being improved by the incorporation of a computer-operated transient digitizer. During the course of testing strong evidence was found that MPITOF mass spectrometry may be a useful technique for studying unimolecular reactions of ions. The evidence is found in the MPITOF mass spectrum of aniline ( $C_6H_5HN_2$ ).

**Reports:**

1. Unimolecular Fragmentation Kinetics by Multiphoton Ionization, by D. Proch et al. *Chem Phys Let.*

**17759 SOME EARLY TRANSITION METALS IN OXIDATION STATE IV USING THE POTASSIUM POLY(PYRAZOL-1-YL) BORATE AS LIGANDS**

Bobby L. Wilson  
Texas Southern University  
SC: CSL

The research objective is to gain insight into the structure, mode of bonding, and magnetic behavior of complexes containing poly(pyrazol-1-yl)borate anions and metals in high oxidation states. The synthesized complexes will be subjected to

characterization using techniques such as electron spin resonance, electronic and vibrational spectroscopy, nmr, and x-ray powder diffraction spectroscopy to gain insight into the structure and mode of bonding. The reaction of hafnium tetrachloride with potassium tetrakis(pyrazol-1-yl) borate in dichloromethane was carried out. These products are currently under study by use of proton magnetic resonance and infrared spectroscopy. The object is to completely characterize the disubstituted and completely substituted products in an effort to better understand the chemistry of hafnium in the plus four state and the ability of the tetrakis(pyrazol-1-yl)borate anion to act as a multidentate ligand.

**17905 NEW SYNTHESIS OF PHOSPHAZENE POLYMERS**

Robert H. Neilson  
Texas Christian University  
SL: AMMRC, NLABS SC: MERADCOM, CSL

The objective of the research is to explore the scope, reaction mechanism, and synthetic generality of the recently discovered route to new phosphazene polymers. The thermal decomposition of suitably constructed Si-N-P precursors is to be used for the synthesis of fully alkyl- and/or aryl-substituted phosphazenes. The new synthesis will be studied as a potential route to many new phosphazenes including several homopolymers, copolymers, and cross-linked polymers.

**17994 LASER CHROMATOGRAPHY (GASEOUS ELECTROPHORESIS OF CHEMICAL AGENTS)**

David M. Luhman  
Quanta-Ray, Inc.  
SL: CSL SC: BRL

The objective is to investigate a new technique termed laser chromatography applicable to the detection of chemical agents. Laser multiphoton ionized molecules in air samples at atmospheric pressure will be separated by diffusion according to their ionic mobilities in nitrogen. A mass spectrometer will be used to provide positive identification of the diffused ions.



# 17997 MULTIPHOTON IONIZATION MASS AND PHOTOELECTRON SPECTROSCOPY

James P. Reilly  
Indiana University

SL: CSL SC: NLABS, BRL, MERADCOM

The research objective is to study the conjunction of laser induced molecular ionization with mass spectrometry and photoelectron spectroscopy. Investigations of the ionization probability of a variety of organic compounds will be conducted. Novel studies of the interaction of laser radiation with isolated gas phase molecules will be carried out to provide molecular spectroscopic, excited state relaxation and ionization potential data.

# 18055 POLYMER CHARACTERIZATION BY ELECTROHYDRODYNAMIC MASS SPECTROMETRY

Kelsey D. Cook  
University of Illinois at Urbana  
SC: MERADCOM, CSI

The research objective is to probe the applicability of electrohydrodynamic ionization mass spectrometry (EHMS) to the characterization of polymer molecular weight averages, distributions, and intrachain impurities. Revision of present EHMS source designs will be tested to improve sample structural information and sensitivity, and to overcome limitations imposed by the need for using solvents of low volatility.

# 18151 THE CONVERSION OF SMALLER BORANE FRAGMENTS TO LARGER STRUCTURES-THE SYSTEMATICS OF BORON HYDRIDES REACTIONS

Robert W. Parry  
Goji Kodama  
University of Utah  
SC: MICOM, BRL

The objective of the research is to explore the reaction of borane addition compounds with  $BH_3$  or other borane fragments to give higher boranes. Studies will be made of reactions involving electron-rich borane adducts (e.g.  $B_2H_4$ ,  $2PMe_3$ , hypho class boranes, etc.), with boranes, and reaction chemistry of weak base adducts of

## B. Organic Synthesis, Organic Mechanisms

boranes, and reactions involving substituted boranes (e.g., haloboranes).

## B. ORGANIC SYNTHESIS, ORGANIC MECHANISMS

# 15618 INTRAMOLECULAR REACTIONS OF UNSATURATED PEROXIDES AND PEROXY RADICALS

N. A. Porter  
Duke University  
SL: NLABS, AMMRC, MERADCOM

The mechanism of the autoxidation of linoleic acid and phospholipid esters of this acid was investigated. The products of autoxidation, 13-hydroperoxy-9, *cis*, 11, *trans*-octadecadienoic (4), 13-hydroperoxy-9, *trans*, 11, *trans*-octadecadienoic (5), 9-hydroperoxy-10, *trans*, 12-*cis*-octadecadienoic (6), and 9-hydroperoxy-10, *trans*, 12-*trans*-octadecadienoic (7) acids were analyzed by HPLC after reduction to the corresponding hydroxy-fatty acids. The ratio of *trans*, *cis*/*trans*, *trans* products (4+6/5+7) formed during the initial stages of oxidation (<2% for the free acids) was dependent on temperature and the concentration of linoleic acid. The *trans*, *cis*/*trans*, *trans* ratio varied from 4.2 (with neat linoleic acid oxidations at 10 degrees) to .23 (.24M linoleic acid in benzene at 50 degrees). A similar product distribution was found in emulsion oxidation of mixtures of 1,2-dilinoyleglycerolphosphatidyl choline and 1,2-dipalmitoylglycerolphosphatidyl choline with the *trans*, *cis*/*trans*, *trans* product ratio depending on the ratio of dilinoylecithin to dipalmitoyl lecithin. Mixtures of linoleic acid and p-methoxyphenol give *trans*, *cis*/*trans*, *trans* product ratios dependent on the concentration of added phenol.

# 15690 THE DESIGN OF FUNCTIONAL AND NON-FUNCTIONAL MICELLES AS DEPHOSPHORYLATING AGENTS

C. A. Bunton  
University of California at Santa Barbara  
SL: CSL SC: NLABS

The dephosphorylation of p-nitrophenyl diphenyl phosphate (PNPDPP) by benzimidazolide or

naphth-2,3-imidazolide ion is catalyzed by both micellized cetyltrimethylammonium bromide (CTABr) and the nonmicellizing phase transfer catalyst, trioctylethylammonium mesylate. Evidence suggests that the phase transfer catalyst forms small aggregates with the reactants, and that the second order rate constant in these aggregates is very similar to that in CTABr micelles. Although phase transfer catalysis is usually carried out in two phase systems there are large rate enhancements of the reactions of hydrophobic nucleophiles in water but reaction with  $\text{OH}^-$  is not catalyzed. Micelles form from CTABr in mixtures of ethylene glycol and aqueous NaOH. Rate enhancements are similar to those in aqueous micelles, but the reactive nucleophile is the alkoxide and not hydroxide ion. Some work has been done on microemulsions of CTABr, octane, *n*-BuOH and aqueous NaOH. The reactive nucleophile is butoxide ion. Quasielastic light scattering is being used to study the properties of these systems. The aggregates seem to have uniform radii, but unexpected changes are evident in diffusion coefficients of the aggregates which are thought to be associated with incipient changes of state rather than structure of the aggregates.

*Reports:*

No. 1-3 in previous edition.

4. Dephosphorylation in Functional Micelles. The Role of the Imidazole Group, by John M. Brown et al, *J Org Chem* 45, 4169 (1980). AD A093 370
5. A Quantitative Treatment of Micellar Effects upon Deprotonation Equilibria, by Clifford A. Bunton et al, *J Phys Chem* 84, 2611 (1980). AD A093 371

**15917 STUDY OF PHOTOPHYSICAL PROCESSES AND MOLECULAR TRANSFORMATIONS OF EXCITED STATES**

H. E. Zimmerman  
University of Wisconsin  
SC: NLABS

A major result has been completion of work on excited state energy dissipation and decay in rod-like molecules. The major thrust of this work deals with modes of energy dissipation with chromophores in varying environments. In continuing research on rod chemistry work has been concerned with methods of obtaining respectable quantities of [4]-rods by coupling two

[2]-rod molecules together. The chief difficulty seems to be the lesser solubility of the [2]-rods compared to the [1]-rods used in the [2]-rod syntheses. This means that the bimolecular concentration of bridgehead free radicals is lower and that the dimerization rate will be less compared to pseudo unimolecular hydrogen abstraction which diminishes the yield. The problem is being dealt with by synthesizing rod-like molecules which have solubilizing long chain polyether and hydrocarbon groups which can be removed subsequently. In further research a study is being made of di-pi-methane systems with a low energy chromophore A and a high energy chromophore B and where the two chromophores must bond as the first step of the di-pi-methane rearrangement. work at this stage has been largely synthetic in preparing a number of different bichromophoric systems. In the picosecond laser studies, work is in progress on methodology for measuring very brief pulses of the order of 1-2 picoseconds.

*Reports:*

No. 1-2 in previous edition.

**16004 GAS PHASE ION-MOLECULE CHEMISTRY OF CARBON, NITROGEN, AND OXYGEN COMPOUNDS**

Charles H. DePuy  
Veronica M. Bierbaum  
University of Colorado  
SC: LCWSL, BRI, MICOM

A comprehensive study was made of the gas phase reactions of alkyl nitrites (RONO where R = methyl, ethyl, *n*-butyl, iso-amyl and neo-pentyl) with a series of anions (amide, hydroxide, fluoride, and the (M-H)<sup>-</sup> ions of acetone, 3-pentanone, 3-methyl-2-butanone, cyclopentanone, cyclohexanone, methylpropionaldehyde, acetonitrile, propylene, cyclohexadiene, pentadiene, propyne, allene, 3-hexyne, 1-pentyne and acrylonitrile). Efforts have continued on investigation of the reactions of anions with nitrogen and oxygen containing compounds and the study of ions with the potential for undergoing highly exothermic reactions. These two themes have come together in a method of generating the previously unstudied acylperoxide ions. Using this

general method of synthesis, it was possible to prepare a number of different peroxyacyl anions including  $\text{CH}_3\text{CO}_2^-$ ,  $\text{CF}_3\text{CO}_2^-$  and  $(\text{CH}_3)_2\text{NCO}_2^-$ . As expected these ions are powerful oxidants. What is unexpected and remarkable is that they are also highly specific in their reactions.

#### Reports:

1. Gas Phase Reactions of Anions with Substituted Silanes, by C. H. DePuy et al, *J Am Chem Soc* 102, 5012 (1980). AD A099 552
2. Direct Detection of the Product Vibrational-State Distribution in the Associative Detachment Reaction  $\text{Cl}^- + \text{H}-\text{HCl}(\nu) + e$ , by Timothy S. Zwier et al, *Phys Rev Let* 44, 1050 (1980). AD A088 862
3. Gas Phase Ion-Molecule Reactions of Alkyl Nitrites and Silanes, by Gary Kenneth King, 1980, 106 p. figures, PhD Thesis.
4. Gas Phase Reactions of Organic Anions as Studied by the Flowing Afterglow Technique, by Charles H. DePuy et al, *Accnt Chem Res.*
5. Gas-Phase Reactions of Cyclic Silanes, by S. A. Sullivan et al, *J Am Chem Soc.*

### 16133 CHEMISTRY ABOVE 10 KBAR

William J. le Noble

State University of New York at Stony Brook

SL: AMMRC SC: BRL, CSL, LCWSL, AFOSR

Work has proceeded in two parallel ways. In one series of experiments, numerous designs were used to overcome the containment problem. This problem, is best described by the statement that in dual ram anvil devices, the sample behaves as though it is under pressure in only the axial direction, and as though there is virtually no pressure at all in the plane perpendicular to the axis. Thus, the sample simply evaporates out of its potassium bromide matrix upon even modest heating, and though the gauge pressure may be 50 kbar or more. This problem is essentially solved, although it is still not completely reliable: some samples still just vanish. The principle being used depends on an ideal combination of properties of gold; which is both strong in the tensile sense, and yet highly flexible. It has a high melting point. The rest of the time has been spent on preparing the chemicals; about five of the more exotic compounds are now available. The trials are being done with similar but simpler and less expensive molecules of about the same volatility.

### 16184 SUPER HYDRIDES

Herbert C. Brown

Purdue Research Foundation

SC: AMMRC, CSL

Recently, borane-methyl sulfide (BMS) has been established to be remarkably stable under refluxing conditions in tetrahydrofuran for 48 h. A means of greatly enhancing reducing properties of borane-methyl sulfide was discovered. Thus, a practical, new reduction procedure for the synthesis of primary alcohols from organic esters in tetrahydrofuran involved heating reactants in the presence of tetrahydrofuran to 85-90°C by using a 9" fractional Vigreux column, allowing dimethyl sulfide to distill off. Organic esters, such as aromatic, aliphatic, cyclic, and highly hindered esters, are selected as representatives. Utilizing a stoichiometric amount of hydride to ester, any organic ester can be readily reduced in 0.5-4 h. An industrial synthetic method for the preparation of lithium borohydride from the readily available and economical reagents, sodium borohydride and lithium bromide, was developed. The procedure adopted was to heat the reactants at 230-240 degrees for 4-8 h without addition of solvent, using a overhead stirrer, followed by addition of ethyl ether to the reaction mixture at 25°C. The reaction proceeded relatively fast at 25°C, requiring 24-36 h for completion. A detailed study of the stepwise reaction of amides with borane-methyl sulfide (BMS) has been made. As a result, an improved method for the rapid reduction of primary amides using a minimum concentration of BMS has been developed. The procedure has been applied to the synthesis of neopentane-diamine dihydrochloride, a useful complexing agent, on a large scale. A systematic study of the reduction of epoxides using super hydride is currently in progress.

#### Reports:

- No. 1-5 in previous edition.
6. Addition Compounds of Alkali Metal Hydrides. 20. Reaction of Representative Mono- and Dialkylboranes with Saline Hydrides to Form the Corresponding Alkylborohydrides, by Herbert C. Brown et al, *J Org Chem.*
  7. Addition Compounds of Alkali Metal Hydrides. 18. Reaction of Trialkylboranes with *t*-Butyllithium. A General, Convenient Method for the Preparation of Lithium Trialkylborohydrides, by Herbert C. Brown et al, *J Organometal Chem* 188, 1 (1980).
  8. Selective Reductions. 27. Reaction of Alkyl Halides with Representative Complex Metal Hydrides and Metal Hydrides. Comparison of Various Hydride Reducing Agents, by S. Krishnamurthy et al, *J Org Chem* 45, 849 (1980).

# BEST DOCUMENT AVAILABLE

## B. Organic Synthesis, Organic Mechanisms

I Chemistry

9. Improved Procedure for Borane-Methyl Sulfide Reduction of Carboxylic Esters, by Herbert C. Brown et al, *Synthesis*.
10. Improved Procedure for Borane-Methyl Sulfide Reduction of Primary Amides to Amines, by Herbert C. Brown et al, *Synthesis*.
11. Improved Procedure for Borane-Methyl Sulfide Reduction of Nitriles, by Herbert C. Brown et al, *Synthesis*.
12. Improved Procedure for Borane-Dimethyl Sulfide Reduction of Tertiary and Secondary Amides in the Presence of Boron Trifluoride Etherate, by Herbert C. Brown et al, *Synthesis*.
13. Exceptionally Rapid Thermal Isomerization of  $\alpha$ -3-Hexyldi-(2, 6-Dimethylcyclohexyl) Borane. A Sterically Enhanced, Highly Efficient Synthetic Route for the Conversion of Internal Acyclic Olefins into Terminal Olefins and Their Derivatives, by Herbert C. Brown et al, *J Org Chem*.

### 16196 CHEMISTRY OF POLYNITROETHANE DERIVATIVES

Kurt Baum  
Fluorochem, Inc.  
SC: MERADCOM

The chemistry of 1, 1, 2, 2-tetranitro compounds is unknown, and the Diels-Alder adducts of tetranitroethylene prepared on this program were used to study such reactions. The anthracene adduct and NaI gave the keto nitronate salt, identified by halogenation. Heating the anthracene adduct under vacuum gave the dinitroolefin, showing that the 1,2 elimination of  $\text{N}_2\text{O}_4$  has some generality. This olefin reacted with NaI and with  $\text{NaNO}_2$  to give the above keto nitronate, with cyclopentadiene to give a Diels-Alder adduct, and with benzylamine to give a zwitterion. Additional work was carried out with the olefin obtained by  $\text{N}_2\text{O}_4$  extrusion from the tetranitroethylene anthracene adduct. Nitro substitution was obtained with sodium butylmercaptide. Spectral evidence was obtained for substitution by sodiummalonate, followed by tautomerization. A manuscript on the chemistry of this olefin is in preparation.

#### Reports:

- No. 1 in previous edition.
2. Synthesis and Reactions of 11, 12-Dinitro-9, 10-dihydro-9, 10-etheneanthracene, by Kurt Baum et al, *J Org Chem*.

### 16201 THE SYNTHESIS OF POLYMERS FOR ELECTRON BEAM AND X-RAY LITHOGRAPHY

Charles U. Pittman, Jr.  
University of Alabama  
SL: ETDL SC: HDL, NVEOL, MICOM

Major results appear in the reports listed below. Chemical composition effects on polymer

absorption of electron beam energy was examined by the use of Bethe's theory for electron scattering in solids. The chemical incorporation of higher atomic-number atoms into the polymer was predicted to decrease energy absorption and increase scattering. This, however, does not allow one to predict the chemistry caused by absorbing the electron energy. Studies have indicated that several fluorine- and chlorine-containing polymer systems give higher chain scission propensities than their unhalogenated analogs. Thus, a broad experimental synthetic program remains necessary (even critical) to the solution of resist-limited problems in electron-beam lithographic approaches to highly density integrated circuit manufacture.

#### Reports:

1. Radiation Degradation Behavior of Chlorine-Containing Vinyl Copolymers. Search for Improved Electron Beam Resists., by John N. Helbert et al, *Polymer Sci and Eng* 20, 630 (1980). AD A095 883
2. Synthesis and Radiation Degradation of Vinyl Polymers Containing Fluorine. Search for Improved Lithographic Resists, by Charles U. Pittman, Jr. et al, *J Polymer Sci* 18, 3413 (1980). AD A095 738
3. Positive Electron Beam Resist Behavior for Methacrylonitrile and Methyl  $\alpha$ -Chloracrylate Polymers and Copolymers, by J. H. Lai et al, *J Vacuum Sci*.
4. Effect of Chemical Composition Upon the Radiation and Electron Beam Resist Behaviors of Vinyl Polymers, by J. N. Helbert et al, *Polymer Sci and Eng*.
5. Synthesis and Polymerization of Methyl  $\alpha$ -Fluoracrylate, by Charles U. Pittman, Jr. et al, *Macromol* 13, 1031 (1980). AD A093 916
6. Synthesis, Radiation Degradation, and Electron Beam Resist Behavior of Fluorine-Containing Vinyl Polymers, by C. U. Pittman, Jr. et al.

### 16318 A STUDY OF CHEMICAL REACTIONS AND INTERACTIONS IN MICRO-EMULSION AND SURFACTANT PHASES

Raymond A. Mackay  
Drexel University  
SL: CSL SC: SLARS

Polarographic measurements of the diffusion coefficients of both oil and water soluble electroactive solutes in O/W microemulsions have now been carried out on a wide range of solute species and emulsion types. A very good semiempirical knowledge has been gained of the behavior of free and weakly bound water soluble



species in both ionic and nonionic microemulsions. Of particular importance are the results using oil soluble solutes. In ionic systems, it was established that the measured diffusion coefficient is actually that of the microdroplet, and from the Einstein equation a droplet diameter can be obtained. This is a very simple measurement to perform, and the results compare favorably with those obtained from low angle x-ray, photon correlation, spectroscopy, and probe fluorescent decay measurements. In nonionic systems, the existence of "percolation" type phenomena previously observed only in W/O systems has been established. Reaction of 2-chlorethyl ethyl sulfide with both borohydride and cyanoborohydride has been observed to occur in a CTAB microemulsion. This is important not only as a new demonstration of enhanced reactivity in microemulsions, but has significant implications for the use of microemulsions as decontaminating and detoxifying solutions since the sulfide reactant is quite similar to sulfur mustard. Investigation of this and similar reactions is continuing. Kinetic studies of the "catalysis" of phosphate ester hydrolysis by hexadecyl pyridinium iodide aldoxime are nearing completion. It is expected that these results will aid in the design of even more effective systems.

#### Reports:

- No. 1 in previous edition.
2. Properties of High Emulsifier Content O/W Microemulsions, by Raymond A. Mackay.
3. Phosphate Ester-Nucleophile Reactions in O/W Microemulsions, by R. A. Mackay et al.

### 16365 A NEW APPROACH TO SUBSTITUTED AROMATICS, POLYPHENYLS, AND CONDENSED AROMATICS

A. I. Meyers  
Colorado State University  
SL: AMMRC SC: NLABS

Studies have continued on non-classical aromatic substitution. Two additional studies are now complete: (1) Metalation of polysubstituted aromatics and (2) annulation of aromatics to polynuclear derivatives. Both have now been published. Further studies on annulations are in progress. Attempts are being made to close the ring

### B. Organic Synthesis, Organic Mechanisms

in a meta-fashion. Polynuclear aromatics are also close at hand and routes to these systems are being explored. Other examples of polynuclear aromatics via methoxyl displacement have led to various lignan lactones.

#### Reports:

- No. 1 in previous edition.
2. Solvent and Base Studies on the Site of Aryl Metalation of 2-(3, 5-Dimethoxyphenyl)-4, 4-Dimethyl-2-Oxazoline, by A. I. Meyers et al, *Tetrahedron Let* 21, 3335 (1980).
3. Nucleophilic Annulations of Aromatics. Novel Route to Benzo-Fused Ring Systems via Oxazoline Activation, by A. I. Meyers, et al, *J Org Chem* 46, 783 (1981).
4. The Chemistry of Aryl Oxazolines. Applications to the Synthesis of Lignan Lactone Derivatives, by A. I. Meyers et al, *J Org Chem*.

### 16382 THE PREPARATION OF FLUOROMETHYLENE OLEFINS

Donald J. Burton  
University of Iowa  
SL: AMMRC SC: NLABS

Work on the  $CX_3/R_3P$  reaction continues. Preliminary work in this area indicates this type of approach will be a general one for homologation of fluoroolefins. This approach is being pursued in more detail. Work on the ylide-carbene reactions has been quite productive as a route to difluoromethylene olefins. A preliminary discovery that it is possible to generate and capture the unstable ylide,  $(RO):P(O)CF_2$ , continues. In addition, it has been found that a stabilized organometallic reagent can be easily produced. This functionalized organometallic reagents is stable (for weeks to months) in solution. Its preliminary reactivity mimics the unstable  $(RO):P(O)CF_2$ . Thus it allows one to use this reagent successfully - without the problems of decomposition of the unstable ylide. This first example of a stable functionalized organometallic phosphonate will be explored in detail.

#### Reports:

- No. 1 in previous edition.
2. Difluoromethylene Chain-Extension Reactions. Preparation of Fluorinated Alkenes and Alkadienes from Olefin Precursors, by Donald J. Burton et al, *J Am Chem Soc* 102, 3980 (1980). AD A096 915



**16391 THE CHEMISTRY OF TRANSITION METAL CARBENE COMPLEXES**

Maurice S. Brookhart  
G. Ronald Husk  
University of North Carolina  
SC: CSI

Synthetic methods have been utilized to expand the list of transition-metal-carbene precursors,  $\text{Cp}(\text{CO})(\text{L})\text{Fe}-\text{CH}(\text{OCH}_3)(\text{R})$ . Successful transfer of the ethylidene fragment to olefins has been achieved. Attempts to transfer ethylidene to other olefins, viz.,  $\text{CH}_2=\text{CH}(\text{CH}_2)_3(\text{CH}_3)_2$ ,  $\text{C}=\text{CHCH}_3$ ; and 2,3-dehydropyran, have been carried out but the studies have not been completed. Similarly, studies of ethylidene transfers are beginning. Neither the ethylidene or propylidene complexes generated can be directly observed by NMR, however, the propylidene complex decomposes rapidly to the well known propene complex by alpha-hydrogen migration. On the other hand, carbene complexes generated at low temperatures can be identified by the characteristic proton resonance. Decomposition pathways have yet to be fully elucidated. Work will continue on alkylidene transfer reactions, as well as resolution of carbene precursors into chiral reagents.

**Reports:**

- No. 1 in previous edition.  
2. Facile Synthesis, Spectral Characterization, and Ethylidene Transfer Reactions of Complexes of the Type  $\text{Cp}(\text{CO})(\text{L})\text{Fe}=\text{CHCH}_3$ , by M. Brookhart et al, *J Am Chem Soc* 103, 979 (1981). AD A098 501

**16527 MACROPOROUS POLYMERIC SUPPORTS FOR ORGANIC SYNTHESIS**

Warren T. Ford  
Oklahoma State University  
SL: NLABS SC: AMMRC, CSI

A method has been developed which uses polystyrene supports to generate ester enolates at ambient temperature and trap them by acylation with little or no interference from self-condensation of the ester. The method has been extended to trapping by alkylation. Both alkylation and acylation of the enolate proceed in  $\geq 95\%$  GC yields and 73-87% isolated yields. The results

demonstrate the utility of a crosslinked polymer support for suppression of self-condensation of esters. The polymer support enables organic synthesis at ambient temperature rather than dry ice temperature as required previously. The spent polymer also has been recycled. Moreover, the work has been performed on a larger scale than most earlier polymer supported synthesis. It can be used to synthesize ester derivatives on a 0.1 mol scale in a 1000 mL flask. The keys to success are (1) use of a more rigid (10% crosslinked) polymer and (2) esterification of the polymer to only partial conversion, which leaves all of the polymer-bound esters in reactive sites in the heterogeneous polymer environment. The performance of ester enolate chemistry in high yield at room temperature at an effective ester concentration of  $\geq 0.1$  M with a recyclable polymer support makes the method highly attractive for testing in industrial syntheses of fine organic chemicals and pharmaceuticals.

**Reports:**

- No. 1 in previous edition.  
2. Carbon-13 NMR Relaxation in Crosslinked Polystyrene Gels, by Warren T. Ford et al.  
3. Acylations and Alkylations of an Ester Enolate in High Yield at Room Temperature on Polystyrene Supports, by Young H. Chang et al.

**16742 STUDIES OF NITRATION AND OXIDATION IN OXYNITROGEN SYSTEMS**

David S. Ross  
SRI International  
SL: MICOM, LCWSL

One goal of this work is to establish the importance of electron transfer in aromatic nitration through the study of the reaction of aromatic radical cations with  $\text{NO}_2$ . Both electrochemical and gas phase ion molecule techniques are being used to study these reactions. For the latter procedure, the aromatic radical cations are formed in a novel combination of photoionization and high pressure mass spectrometry procedures. Perrin has reported that the reaction of anodically generated naphthalene radical cation with  $\text{NO}_2$  in acetonitrile yields nitronaphthalenes in the ratio  $\alpha:\beta = 9 \pm 1$ . Ebersson et al. have more recently cast some doubt regarding this study; they obtained a different isomer ratio for the product nitronaphthalenes,

$\alpha:\beta = 20 \pm 3$ , and suggested that the process is an electrochemically initiated, homogeneous reaction of naphthalene with trace  $\text{HNO}_2$  or  $\text{HNO}_3$ . To resolve the discrepancy and to study the effect of small amounts of water (which according to Perrin were responsible for the changes in the isomer ratio), similar electrochemical nitrations were conducted. Ebersson's observation was confirmed that in  $\text{CH}_3\text{CN}$ , controlled potential electrolysis of naphthalene results in  $\alpha$ - and  $\beta$ -nitronaphthalenes in the ratio  $20 \pm 3$ .

## Report:

1. Catalysis of Aromatic Nitration by the Lower Oxides of Nitrogen, by David S. Ross et al, *J Chem Soc Chem Commun* p532, 1980. AD A091 471

### 17283 THE CHEMICAL PRODUCTION OF EXCITED STATE MOLECULES

William H. Richardson  
San Diego State University  
SL: NWC, NLABS

The research objective is to study electron transfer processes involved in chemical production of the excited states in dioxetanes and diacetyl peroxides leading to chemiluminescence. An attempt will be made to correlate molecular structure with excited state efficiency. The effect of substituents on triplet efficiencies in the series of 3, 4-diaryl-3, 4-dimethyl-1,2-dioxetanes will be studied. Lifetimes of key reaction intermediates will be determined. New chemiluminescent systems will be designed and synthesized.

## Reports:

1. Kinetics and Mechanism of the Thermolysis of a Five-Membered Ring Peroxide. 3, 3, 5, 5-Tetramethyl-1, 2-dioxolane, by William H. Richardson et al, *J Org Chem* 46, 1887 (1981).
2. Haloaromatic Substituted Olefins by the McMurry Olefin Synthesis, by William H. Richardson, *Synthetic Commun.*

### 17572 IPSO NITRATION, REGIOSPECIFIC NITRATION VIA IPSO NITRATION PRODUCTS

Philip C. Myhre  
Harvey Mudd College  
SL: LCWSL, AMMRC SC: MICOM

An intensive investigation of the ipso-nitration reaction of aromatic compounds will be carried

out. Reaction sequences permitting regio specific nitration of alkylaromatic compounds via ipso nitration will be investigated. Both exploratory studies and pertinent mechanistic investigations will be carried out. Finally, optimization of established procedures will be established. The preparation of the ten possible 4-methyl-4-nitrocyclohexadienones that bear varying numbers of methyl groups at other ring carbons is nearing completion, and kinetic evaluations of the thermal stability of these compounds are underway. Crossover studies with doubly labeled 4-methyl-4-nitrocyclohexadienones in fluid and viscous non-polar solvents and in various concentrations of sulfuric acid have been conducted. Work has progressed on synthetic methods for synthesis of 1, 4-dialkyl-4-nitrocyclohexadienols by addition of alkyl lithium reagents to 4-alkyl-4-nitrocyclohexadienones. Present work is directed toward the synthesis of several specifically labeled, 1, 4-dimethyl-4-nitrocyclohexadienols.

### 17579 PREPARATION OF NITROCOMPOUNDS

George A. Olah  
University of Southern California  
SL: LCWSL, MICOM SC: NWC

An exploration into new methods of preparation of nitro and polynitro-compounds will be carried out. Several new nitrating agents will be studied to effect the polynitration of benzene and its derivatives, these include the mixed "fluoroacid"  $\text{FSO}_3\text{H}-\text{HNO}_3$ ; mixed anhydrides of nitric acid with trifluoromethane-sulfonic acid, fluorosulfonic acids, and selected carboxylic, sulfonic, and carboxylic acids; nitroammonium salts, N-nitroimidazole and pyrazole, and nitratotri-methoxyphosphonium salts. Novel solvent systems and techniques will also be employed.

### 17582 DINITROACETYLENE AND RELATED COMPOUNDS

Michael J. S. Dewar  
University of Texas  
SL: MICOM, LCWSL, SC: NWC, NSWC  
FJSRL CSL, MICOM

A study will be made of dinitroacetylene and its derivatives. A series of preparative methods will be

used to effect the synthesis of dinitroacetylene and related compounds. Polynitro compounds resulting from oligomerization of nitroacetylenes will also be investigated in regard to physical and chemical properties.

**17777 REACTIVITY OF  
FERROCENYLPHOSPHINE  
DERIVATIVES TOWARD  
NUCLEOPHILIC DISPLACEMENT**

Arnold W. Smalley  
Southern University at Baton Rouge  
SC: CSL

The stabilizing effect of ferrocenyl substituents on a neighboring positively charged species will be studied. A series of ferrocenyl-substituted quaternary phosphonium salts containing various electron-donating and withdrawing groups on the ferrocenyl group will be synthesized. The effects of the substituents on the stability of the phosphonium salts will be determined. Attention has been directed toward (1) purification of solvents to be used in the initial research, (2) procuring supplies and equipment, and (3) initial study of the synthesis of aryl-substituted ferrocenyl-phosphines.

**17858 THE SYNTHESIS AND ORGANIC  
CHEMISTRY OF THE BORON-  
SILICON BOND**

Gerald L. Larson  
University of Puerto Rico

The synthesis, characterization, and reactions of organometalloid compounds containing the boron-silicon bond will be undertaken. Synthesis of compounds containing the boron-silicon bond will be attempted utilizing silyl-lithium compounds, chlorosilyllithium reagents, desilaboration reactions, and other interesting approaches. The resultant compounds will be characterized spectroscopically. Chemical properties will be established in relation to unsaturated species and towards oxidation and hydrolysis.

**17899 HIGHLY REACTIVE METAL  
POWDERS**

Reuben D. Rieke  
University of Nebraska at Lincoln  
SL: AMMRC SC: LCWSL, CERL

The chemical and physical properties of metal powders resulting from the reduction of metal salts in organic solvents will be delineated. Highly reactive metal powders will be prepared by reduction of the corresponding metal salts. The chemistry of these metals will be explored in a variety of oxidative-insertion reactions, towards formation of zerovalent derivatives, and in catalytic reactions.

**17986 THE CHEMISTRY OF OPTICALLY  
ACTIVE MACROCYCLIC  
POLYETHERS**

Richard A. Palmer  
Robert G. Ghirardelli  
Duke University  
SC: CSL

(1) Investigation of exchange rates for crown ethers complexed to alkali metal ions: The rate of complexation of the  $Mg^{2+}$  cation by 12-crown-4 in acetonitrile solution was studied using NMR spectroscopy. The rate of exchange between the free and complexed ligand is slow on the NMR time scale at room temperature. The temperature dependence of the rate was followed by a comparison of computer simulated spectra to spectra obtained for a series of temperatures from the point of slow exchange to the coalescence point of the two signals (fast exchange). Although complete coalescence was not observed, the computer line shape fit indicates an exchange rate varying from  $0.5 \text{ sec}^{-1}$  at  $25^\circ\text{C}$  to  $33 \text{ sec}^{-1}$  at  $75^\circ\text{C}$ . An analogous exchange has also been shown to occur with the  $Li^+$  cation and 12-crown-4. (2) Investigation of exchange equilibria of alkali metal salts in chloroform: The use of chiral crown ethers to complex alkali metal salts in such relatively nonpolar solvents as chloroform has allowed the investigation of exchange phenomena by the use of circular dichroism. Anion exchange between a crown-ether complexed alkali metal ion and some

other non complexed cation can be monitored providing one of the anions in the process is an accessible chromophore. Investigations thus far have led to the calculation of the exchange equilibrium constants of two reactions.

#### 18005 CATALYTIC OXIDATION BY TRANSITION METAL IONS IN ZEOLITES

Jack H. Lunsford  
Texas A&M Research Foundation  
SL: CSL SC: CSL, USATHAMA

The objective of the research is to understand oxidation reactions in zeolites at a molecular level and determine superior catalysts for certain oxidation reactions. A systematic investigation of selected oxidation reactions will elucidate the elementary processes occurring within the cavities of a zeolite. Kinetic and spectroscopic techniques will be used to follow reaction intermediates and stable products. Radical intermediates will be detected by epr.

#### 18090 SYNTHESIS AND REACTIONS OF LARGE GLOBULAR MOLECULES

G. David Mendenhall  
Michigan Technological University  
SC: LCWSL, NLABS, AMMRC

The reactions of functional groups attached to large, globular hydrocarbon molecules will be investigated with the aim of developing new synthetic methods. Tri-*tert*-butylacetic acid and its derivatives will be used to prepare functionalized, higher-rank, condensed structures. The resultant globular molecules will be used to prepare persistent free radicals, organometallics, and unusually selective organic reagents. Large globular molecules with attached polar groups will be tested as 1-component micelles as well for their permeability and enhanced volatility.

#### C. STRUCTURAL CHEMISTRY, SURFACE CHEMISTRY, AND SPECTROSCOPY

##### 15521 ENERGY LEVELS IN AND ON AN OXIDE FILM AS MEASURED BY ELECTROCHEMICAL METHODS

S. R. Morrison  
SRI International  
SL: ETDL SC: CSL, MERADCOM

A simple method based on the linear relationship between experimental metal work functions and Pauling electronegativities has been used to obtain effective solid state Fermi energies for atoms, both metallic and nonmetallic. A simple arithmetic mean combination rule was used to give the intrinsic Fermi level of a solid, and this level was then combined with band gap data to give electron affinities. The method was applied to materials covering a wide range of band gap and ionicity. The results were in good agreement with literature values of electron affinities. Electron flow between silicon and an aqueous solution through a thermally grown SiO<sub>2</sub> layer has been observed after a suit ble activation process. The activation is either a heat treatment in vacuum or a negative bias applied to the silicon. The duration required for the cathodic bias is consistent with the time needed to provide a high positive ion density in the SiO<sub>2</sub>. As the SiO<sub>2</sub> becomes more electrically conducting, copious hydrogen evolution begins. If metal ions such as zinc are present they can be reduced giving metal deposits. If sufficiently high voltage is applied, cathodic luminescence, characteristic of the ions in solution, is observed. When the electronic conductivity is obtained, the system Si/SiO<sub>2</sub> behaves like a metal electrode, in that current is passed to reducing or oxidizing agents in solution when the electrode potential reaches the E degree for the redox couple. The results can be explained in terms of field-aided ion diffusion into the oxide under the cathodic bias, occurring particularly at flaws in the oxide.

##### Reports:

No. 1-4 in previous edition.

##### 15703 FLUORESCENCE AND LIGHT SCATTERING OF AEROSOL PARTICLES

Milton Kerker  
Clarkson College of Technology  
SL: CSL SC: ASL, NVEOL

Flourescent and Raman scattering by molecules in dielectric particles is strongly dependent on the



morphology and optical properties of the particle, the distribution of active molecules within the particle, and, in the case of nonspherical particles, orientation. The model previously applied to spheres and cylinders has been extended to spheroids. The extended boundary condition method (EBCM) has been used to calculate the transmitted field at the incident frequency that stimulates the process. The equivalence principle underlying the EBCM has also been applied to calculate the fields at the shifted frequency. Numerical results illustrate some of the effects of refractive index, size, shape, and orientation of the particles for models representing two polarizabilities of active dipoles embedded inside the particle. A paper has been published in which a model for Raman scattering by a molecule adsorbed at the surface of a spherical particle is articulated by treating the molecule as a classical electric dipole. This follows Moskovit's suggestion and the experiments by Creighton, et al that such a system may exhibit SERS similar to that at roughened electrode surfaces. The molecule is stimulated by a primary field comprised to the incident and near-scattered fields. Emission consists of the dipole field plus a scattered field, each at the shifted frequency. Addition of feedback terms between the dipole and the particle makes only a negligible contribution to the fields. In another paper, experimental Raman signals, enhanced greater than  $10^5$ -fold are reported from citrate ion adsorbed on colloidal silver. Although this confirms the recently formulated theoretical model in that SERS comparable to that observed on roughened macroscopic surfaces, also occurs in colloidal dispersions, the measured wavelength dependence of Raman intensity differs from that predicted by the theoretical model. The most important point to note at this time is that these large Raman band enhancements of species adsorbed at the surface of silver colloids permit structural and kinetic studies similar to those at roughened surfaces. Colloidal sols, as a substrate have a number of advantages.

*Reports:*

- No. 1-5 in previous editions.  
6. Surface Enhanced Raman Scattering (SERS) of Citrate Ion Adsorbed on Colloidal silver, by M. Kerker et al, *Appl Opt* 19, 3253 (1980), AD A095 739

**16273 DETERMINATION OF THE MICROSCOPIC STRUCTURE OF SURFACES AND OVERLAYERS, ABSORBATE-ADSORBATE INTERACTION ENERGIES, AND THE RATE OF SURFACE PROCESSES**

W. Henry Weinberg  
California Institute of Technology  
SL: ICWSL, MERADCOM SC: BWL, BRL

Insofar as work in low-energy electron diffraction is concerned, an excluded area model has been formulated to describe the relative positions of ordered islands on a crystal surface. Studies have indicated that the kinematically diffracted intensity depends solely on the distribution of island sizes, and, using this result, the intensity and half-width of the LEED beams have been calculated as functions of surface coverage for one model of island growth. A great deal of effort has been devoted toward the design and construction of a new molecular beam-surface scattering machine that will allow the measurement of reaction rate coefficients of corrosive gases interacting with well characterized surfaces of iron. All aspects of the design have been finalized, and all major items or equipment have been ordered.

*Reports:*

1. Intensities of Low-Energy Electron Diffraction Beams for Arrays of Ordered Islands, by Ellen D. Williams et al, *Surface Sci.*

**16951 THE NATURE AND PHOTOCHEMISTRY OF ORGANIC MOLECULES ADSORBED ON CLAYS**

J. Kerry Thomas  
University of Notre Dame  
SL: CSI SC: NLBS

The major research area is photochemistry in colloidal clay systems. In order to understand the details of photochemistry in these complex systems, photochemistry has been studied in model systems such as colloidal silica particles, and anionic perfluoromicelles. All systems share common features i.e. little affinity for neutral molecules in the unaltered state, and strong affinity for cationic species. The silica particles and clays contain identical anionic binding groups ( $\text{SiO}^-$ ), while the



perfluoromicelles provide a linkage between the chemistry of silica particles and conventional anionic micelles. Many of the ideas associated with photochemistry in micelles may be carried over into colloidal silica and clay systems. Additional new features are found in the latter groups of systems in particular with respect to specific aggregation of organic entities on these surfaces.

**17080 CHEMICAL AND PHOTOCHEMICAL REACTIONS OF PORPHYRINS AND METALLOPORPHYRINS IN MICELLES, VESICLES, FILMS, MONOLAYER ASSEMBLIES AND SOLUTION**

David G. Whitten  
University of North Carolina  
SC: LCWSL, NLABS

The objective of the research is to enhance the understanding of the metallation of porphyrins in micelles, vesicles, monolayer assemblies and in solution. Rate studies will be carried out utilizing absorption and fluorescence spectroscopy; intermediates formed on photolysis and photooxidation will be isolated and identified; the ability of ionophores and other phase transfer agents to facilitate metallation will be measured. Protoporphyrin IX (1) as well as a number of other porphyrins are excellent sensitizers for photooxidations. Protoporphyrin IX will also photooxidize itself to give two isomers of photoprotoporphyrin IX (2) as major products. Initial studies in this area indicated that other products are also formed in the photooxidation including both isomers of monoformyl, monovinyl deuteroporphyrin and diformyldeuteroporphyrin. Studies have focused mostly on the solution photochemistry of 1 and the solvent effect of product 2 and the aforementioned formyl products. Two new cobalt complexes have been synthesized which are related to the photochemically labile cobalt(III)  $(\text{NH}_3)_5 \text{X}^{2+}$  complexes which have previously been well studied. These complexes, of course, are known to give fairly reactive halogen radicals on excitation in the ultraviolet; the surfactant analogs which have been synthesized in fact show the same reactivity as their nonsurfactant analogs in solution photochemical studies. Studies have indicated that the surfactant

compounds form stable monolayer films at the air-water interface, and that these films can be readily transferred to build up supported multilayer assemblies. The complex in arachidic acid-complex mixed layers shows an absorption spectrum almost identical to that obtained in solution.

**17147 CARBON-SUPPORTED IRON AS CARBON MONOXIDE AND BENZENE HYDROGENATION CATALYSTS**

M. Albert Vannice  
Pennsylvania State University  
SL: MERADCOM SC: MERADCOM

The research objective is to prepare doped glassy carbons, to analyze the effect of the dopants on the electron concentration in the carbon and on the electronic properties of the iron dispersed on the carbon surface, and to characterize the catalytic properties of these iron/carbon systems. The techniques of x-ray diffraction, EPR and Mössbauer spectroscopy, and magnetization measurements will be used to characterize the iron crystallites, the electronic state of the carbon supports, and the chemical (electronic) state of the iron crystallites. *Sample Preparation:* Two different series of carbon catalyst supports have been prepared using Monarch 700 and Monarch 880 carbon blacks. A portion of Monarch 700 was heat treated in Ar to 2773K to polygonize (graphitize) the particles to some degree. Boron-doped (1 wt %) Monarch 700 and graphitized 700 blacks were prepared by aqueous impregnation of boric acid followed by drying. Samples of each of the B-loaded carbons were heated in Ar to both 2073 and 2773K. The objective was to diffuse boron substitutionally into the carbon lattice and thereby lower the Fermi level (decrease the electron concentration) in the carbon. *Electronic Properties:* Magnetic susceptibility measurements at 300K on boron-doped samples were carried out using a modified Faraday method. The heat-treated Monarch 700 had an average magnetic susceptibility per gram near that of graphite while, as expected, the amorphous 700 had a much smaller value. In all cases, the addition of boron decreased the diamagnetism with the largest effect occurring with the heat-treated 700. Similar results were obtained after hydrogen reduction at 673K. These results clearly establish the electron-acceptor role of boron.

**17349 INTERACTIONS OF VIBRATIONALLY EXCITED MOLECULES WITH SOLID SURFACES**

Robert P. Merrill  
Paul L. Houston  
Cornell University  
SC: BRL, LCWSL

The objective of the research is to investigate collisions of vibrationally excited gas molecules with well-defined solid surfaces in order to examine surface induced de-excitation of molecular vibrations and the effect of vibrational excitation on the reaction cross-sections of gas-solid chemical reactions. Vibrational de-excitation will be determined by measuring the decay in fluorescence. Reaction cross-sections will be measured mass spectrometrically. Preliminary results on the relaxation of CO<sub>2</sub> at a clean stainless steel surface have been obtained. Molecules contained in an UHV chamber were excited with a q-switched CO<sub>2</sub> laser. Their concentration was then monitored as a function of time by observing their vibrational fluorescence, through an infrared detector. Under the conditions of the experiment, vibrational relaxation was controlled primarily by collisions of the excited molecules with the walls of a cylindrical container constructed of stainless steel. The exponential decay rate of the fluorescence signal was extracted to obtain information about the efficiency of vibrational deactivation per gas-surface collision.

**17355 STUDY OF GAS-SURFACE INTERACTIONS BY LASER-INDUCED FLUORESCENCE DETECTION**

Richard N. Zare  
Stanford University  
SL: LCWSL SC: AMMRC, BRL

The research is to investigate the mechanisms of gas-surface interactions by conducting experiments which will simultaneously observe distributions of vibrational, translational, and rotational energy and orientation. These variables will be measured as a function of the scattering angle of the emitted molecule with respect to the surface, and the angle, velocity, and internal temperature of the incident beam. The temperature and crystal face of the surface will also be varied, and scattering can be observed from an adsorbed layer. Elementary gas surface interactions will be studied through

coupling laser induced fluorescence with ultra high vacuum techniques in order to observe the motion of molecules which have just left a single-crystal surface. Laser induced fluorescence has been used to measure the rotational state distribution of nitric oxide scattered from clean, single-crystal silver. Each resulting distribution can be described by a rotational temperature within the statistical uncertainty. As the surface temperature is increased from 475 to 615 to 720K, the calculated rotational temperature varies from 360  $\pm$  50K to 430  $\pm$  90K to 405  $\pm$  60K, respectively. This shows that not only is rotational accommodation incomplete, but the degree of accommodation may decrease at the highest surface temperature studied.

*Reports:*

1. Determination of Internal State Distributions of Surface-Scattered Molecules: Incomplete Rotational Accommodation of NO on Ag(111), by Gray M. McClelland et al, *Phys Rev Let.*
2. Stepwise Bond Dissociation Energies for the Removal of Fluorine from Thionyl Fluoride and Sulfuryl Fluoride, by Teddy Kiang et al, *Chem Commun*, 1228 (1980). AD A099 544

**17451 MODELING THE INFRARED REFLECTANCE AND EMITTANCE OF PAINTS AND COATINGS**

James R. Aronson  
Arthur D. Little, Inc.  
SL: MERADCOM SC: NLABS, MERADCOM, WADC

The research objective is the investigation and development of modeling capabilities for the reflectance of paints and to investigate methods for obtaining accurate values of optical constants for the various components of paints. Computer simulation techniques will be used with available optical constants and values of constants yet to be determined of various pigments and binders in order to model the reflectance and/or emittance properties of paints.

**17464 FLUORESCENT GUESTS IN THERMOTROPIC AND CHIRALITY IN LYOTROPIC LIQUID CRYSTALS**

Mortimer M. Labes  
Temple University  
SL: ETDL SC: CSL

The research objectives are (1) to determine the factors governing the emissive and absorptive

properties of guest molecules in liquid crystal hosts as a function of the former's molecular size, shape and polarity, and (2) to determine the nature of the interaction between chiral guests and chiral lyotropic phases, and measure the kinetics of reactions undergone by the guests. Spectroscopic techniques will be employed to measure the absorption and reflection of light, as well as the emission when fluorescent guest molecules are introduced into liquid crystal systems.

#### 17861 THE PHASE TRANSFER CATALYZED HYDROLYSIS OF ALKYL AND ACYL HALIDES

H. Dupont Durst  
University of Puerto Rico  
SC: CSL

The research objective is to establish the basic chemical principles needed to design an effective cocatalytic phase transfer system for the hydrolysis of a broad class of alkyl and acyl halides. Various lactones and halide compounds will be synthesized to serve as substrates, and different phase transfer catalysts will be evaluated for their ability to enhance rates of hydrolysis. Support of the catalysts on polymers will be effected.

#### 17911 CHEMICAL INTERACTION OF GASES WITH ACTIVATED CARBON USING FOURIER-TRANSFORMED INFRARED PHOTOACOUSTIC SPECTROSCOPY

Mark Rockley  
Oklahoma State University  
SC: CSL, MERADCOM

The research objective is to examine the chemical interaction between simple gases and activated carbon. The technique of Fourier-transformed infrared photoacoustic spectroscopy will be used. Since the method is sensitive to absorption by fractions of an absorbed monolayer, it is expected that information on molecular fragments and the strength and type of the chemisorptive bond can be obtained.

#### 17933 MICELLE FORMATION UNDER UNUSUAL CONDITIONS & OPTIMIZING SOLUBILIZATION IN MICELLAR SOLUTIONS

D. Fennell Evans  
University of Minnesota  
SC: CSL, USATHAMA

The research objectives are to characterize micelle formation in low melting fused salts and compare with the situation in water, and to determine rates of solubilization of liquids in micellar solutions. Vapor pressure, surface tension, conductance, calorimetric measurements will be carried out. Critical micelle concentrations will be determined from nuclear magnetic resonance and light scattering. A spinning disc device will be used to measure solubilization kinetics.

#### D. KINETICS AND THERMODYNAMICS

#### 14081 NANOSECOND AND PICOSECOND SPECTROSCOPY AND KINETICS OF DYNAMIC ABSORBING MATERIALS

M. W. Windsor  
Washington State University  
SL: NLABS SC: CSTA, CSL, MICOM

Studies in micellar media of fast absorbance changes arising from photo-induced electron transfer between porphyrin donors and quinone acceptors have continued. A technique has been developed for automatic acquisition of broad-band transient difference spectra with only a few laser shots. The detection system uses a standard vidicon-optical multichannel analyzer combination interfaced to a reliable, home-assembled microcomputer. The combination is very cost effective and provides difference spectra over a range of 300 nm with excellent resolution in both absorbance and wavelength. Lower resolution spectra, suitable for exploratory study of new systems are obtained in a matter of minutes with only a few laser shots - a great advantage in the case of samples susceptible to photodegradation. A joint study is being made with Dophine's group (UBC, Vancouver) and Gouterman's group (UV, Seattle) of the electronic spectra of a series of etioporphyrin dimers joined by a single  $(CH_2)_n$  bridge of variable length ( $n = 0$  to 8).

## Reports:

- No. 1-3 in previous editions.
4. Picosecond Studies of Primary Charge Separation in Bacterial Photosynthesis, by M. W. Windsor et al, *Phil Trans Roy Soc London A* 298, 335 (1980).
  5. A Study of the Viscosity-Dependent Electronic Relaxation of Some Triphenylmethane Dyes Using Picosecond Flash Photolysis, by David A. Cremers et al, *Chem Phys Let* 71, 27 (1980).
  6. Electron Transfer from Photoexcited Bacteriopheophytin to p-Benzoquinone in Cationic Micelles, by D. Holten et al, *Picosecond Phenomena II*, ed by R. Hochstrasser et al, p317, Springer-Verlag, 1980.
  7. Picosecond Studies of Electronic Relaxation in Triphenylmethane Dyes by Fluorescence-Upconversion, by G. S. Beddard et al, *Picosecond Phenomena II*, ed by R. Hochstrasser et al, p167, Springer-Verlag, 1980.
  8. Automated Picosecond Spectroscopy with a Standard Two-Dimensional Optical Multichannel Analyzer and an Inexpensive Microprocessor, by Dewey Holten et al, *Photobiophys* 1, 243 (1980).
  9. Models for Photosynthetic Charge Separation: Excited State Electron Transfer reactions of Bacteriopheophytin in Micelles, by S. Sadiq Shah et al, *Photobiophys* 1, 361 (1980).
  10. Ring Interactions in Covalently-Linked Porphyrin Dimers, by Ron Selensky et al, *Chem Phys*.

# 15557 REACTIONS OF ELECTRONICALLY EXCITED ALKALINE EARTH ATOMS WITH SMALL MOLECULES

P. J. Dagdigan  
Johns Hopkins University  
SL: BRL SC: MICOM

An investigation of the chemiluminescent reaction of metastable Ca atoms with N<sub>2</sub>O has been completed. By variation of the atom source conditions, it is possible to separate the contributions due to Ca <sup>1</sup>S, <sup>3</sup>P°, and <sup>1</sup>D reactant states. The chemiluminescence has also been investigated up to pressures as high as 0.3 Torr. The yield of CaO A<sup>1</sup>Σ<sup>+</sup> excited molecules was determined to increase significantly with N<sub>2</sub>O pressure, indicative of facile collisional energy between nonemitting and emitting excited CaO\* molecules. More detailed studies are being made of the chemiluminescence of metastable Mg and Sr atoms with N<sub>2</sub>O. The latter reaction yields strong visible and near infrared emission, in qualitative agreement with the Ca results. In addition, there also appears to be collisional energy transfer processes occurring in SrO\* at higher pressures. By contrast, the Mg<sup>3</sup>P° + N<sub>2</sub>O reaction produces only barely detectable luminescence. This result has

implications for the reported use of this reaction as an indicator of the presence of Mg\*(<sup>3</sup>P°) in Mg/CO/N<sub>2</sub>O flames. Other efforts include (1) a laser-fluorescence study of the Mg(<sup>3</sup>P°) + <sup>1</sup>O<sub>2</sub> → MgO' + <sup>1</sup>O reaction, with a goal of determining the MgO product electronic state branching ratio and comparing it with theoretical predictions, and (2) developing theoretical models for the collisional processes, subsequent to initial formation of alkaline earth oxides, which can act to transfer population from nonradiating excited electronic states into the radiating state.

## Reports:

- No. 1-5 in previous editions.
6. Validity of Energy Gap Representations of Rotationally Inelastic Cross Sections Between Polar Molecules, by Millard H. Alexander et al, *J Chem Phys* 73, 3797 (1980). AD A099 658
  7. Collisional Energy Transfer Between Dark and Radiating Excited Electronic States: Steady State Model for CaO-(A<sup>2</sup>Π) + N<sub>2</sub>O ⇌ CaO(A<sup>1</sup>Σ<sup>+</sup>) + N<sub>2</sub>O, by Millard H. Alexander, *J Chem Phys*.
  8. A Study of the Reactions Ca(<sup>1</sup>S, <sup>3</sup>P°, and <sup>1</sup>D) + N<sub>2</sub>O Under Single-Collision Conditions and at higher Pressures: Chemiluminescence Cross Sections, Photon yields, and Collisional Energy Transfer in CaO by N<sub>2</sub>O, by John A. Irvin et al, *J Chem Phys* 74, 6178 (1981).

# 15591 FUNDAMENTAL CHEMICAL PROCESSES IN EXPLOSIVES INITIATION

D. F. McMillen  
D. M. Golden  
SRI International  
SL: LCWSL BRL SC: BRL MERADCOM, NSWC, ONR

The recent accomplishments and current status of the effort to modify Laser-Powered Homogeneous Pyrolysis (LPHP) for use with low vapor pressure, surface-sensitive compounds are: (1) Further measurements of IR radiation from indirectly heated CO, which indicate that changing the geometry of the cell and the path of the laser beam within a given cell markedly affect the pattern of reflected shocks and the temperature-time profile. (2) Self-consistent LPHP measurement of Arrhenius parameters in an over-determined system consisting of three substrates that react by molecular (non-radical) pathways, and for which Arrhenius parameters were already well established. (3) Successful establishment of conditions under which radicals generated by bond scission reactions of ethylbenzene are



quantitatively scavenged and the known absolute rate is reproduced. (4) In experiments currently underway, temperature is being varied in order to obtain the activation energy for bond scission. When the scavenging has been shown to be effective for ethylbenzene as substrate over the range of temperatures required, ortho- and para-nitrotoluene will be used as substrates.

#### 15826 EXPERIMENTAL AND MODELING STUDIES OF HIGH TEMPERATURE COMBUSTION

Reinhard Zellner  
University of Texas at Austin  
SL: ARRADCOM SC: BRL, MICOM

Ignition delay times in air for hydrocarbon mixtures that simulate natural gas were measured following the shock heating of such mixtures in reflected shock waves. The onset of ignition was determined by time-resolved absorption of the 3.39  $\mu\text{m}$  He-Ne laser line. Measured induction times ranged from 20-1200  $\mu\text{s}$  over the temperature interval 1200-1850K at total densities near  $3 \times 10^{-5}$  mol/cm<sup>3</sup>, corresponding to a pressure of about 4 atm at 1600K. Ignition delays were measured for 1% CH<sub>4</sub>, 0.1% C<sub>2</sub>H<sub>6</sub>, and C<sub>3</sub>H<sub>8</sub>, by the ir laser-absorption method over the temperature 1300-1800K and densities near  $2.5 \times 10^{-5}$  mol/cm<sup>3</sup>. In supporting experiments the thermal dissociation rate of C<sub>3</sub>H<sub>8</sub> was measured for similar conditions by the same technique. Computer modeling was carried out to interpret both sets of results.

Reports:  
No. 1-8 in previous editions.  
9. Acceleration of Methane-Air Ignition by Hydrocarbon Additives, by C. S. Fubank et al.

#### 15879 INVESTIGATIONS OF CHEMILUMINESCENT REACTIONS

A. P. Schaap  
Carl R. Johnson  
Wayne State University  
SL: ICWSL SC: NLABS, MERADCOM

Investigations of the catalytic decomposition of 1,2-dioxetanes are continuing. Both silica gel and

#### D. Kinetics and Thermodynamics

CF<sub>3</sub>CH<sub>2</sub>OH are effective in catalyzing the decomposition of dioxetane with greatly enhanced chemiluminescence efficiencies. Additional catalysts such as substituted phenols and carboxylic acids are being studied. A long range goal of this research is the development of an efficient, practical chemical light source. Investigations of electron transfer photooxidation are continuing. The first example of the photooxidation of a cyclopropane has been observed using 9,10-dicyanoanthracene (DCA) sensitization. Irradiation of a solution of tetraphenylcyclopropane and DCA in acetonitrile under O<sub>2</sub> yields the cyclic peroxide. ESR experiments to observe the intermediate cation radical are planned. In the area of chemiluminescence, a continuing problem has been the absolute calibration of the sensitivity of the chemiluminescence measuring instruments used by various research groups. Two liquid light standards have been used to date: (1) the chemiluminescent reaction of luminol and (2) the visible light emission from <sup>14</sup>C-radioactive source in the presence of a scintillation cocktail. However, considerable difficulties remain with the use of these standards and chemiluminescence quantum yields based on both standards often differ by a factor of 2-3. As an approach to solving this problem, a liquid light standard based on a luminescent dioxetane is being developed.

Reports:  
No. 1-4 in previous editions.

#### 16017 STRUCTURES AND INTERACTIONS IN ORGANIC AGGREGATES

Richard H. Clarke  
Boston University  
SL: NLABS

The focus of the work has been on the organic intramolecular dimer, a case expected to probe the theory of triplet excitons applied to organic aromatic systems in the extreme limit of strong coupling (viz., the interaction between the two monomer units of the dimer is due to the presence of a covalent bond). In addition to the biquinoline system, the molecules which began this series, efforts have concentrated on the triplet state ODMR spectrum of *trans* binaphthyl and tribenzotriptycene (trinaphthyl, basically). The work on biquinoline has progressed to the point



where true dimer states are being observed in the biphenyl host matrix, i.e. population of both the symmetric and antisymmetric dimer levels in the triplet and the subsequent magnetic resonance transitions at zero field in both levels. This is an extremely important result in dimer spectra, since it allows measurements of both coherence times, exchange rates and exchange splittings, to be described in terms of dimer theory for a single molecule. Work on synthesis of tribenzotriptycene has progressed to the point where fully characterized emission spectra are now being obtained. With these spectra, ODMR experiments can be initiated, when appropriate solvents and mixed crystal hosts have been determined.

#### Reports:

- No. 1-3 in previous editions.
4. Phosphorescence and Zero-Field ODMR of Biquinoline, by R. H. Clarke et al, *Chem Phys Let* 76, 237 (1980).
  5. Electron Delocalization in the Lowest Triplet State of the Covalently-Linked Pyrochlorophyllide *a* Dimer, by Robert F. Clark et al, *J Chem Phys* 73, 5386 (1980).

### 16110 INVESTIGATION OF GAS-PHASE REACTIONS OF IMPORTANCE IN IGNITION PROCESSES

S. W. Benson  
University of Southern California  
SL: MERADCOM, BRL SC: LEWIS

The ion source of the mass spectrometer has been redesigned so that the high sensitivity of the original design can be used but with improved electron beam energy control which will allow one to measure radicals directly at low concentration with large parent back grounds. A comprehensive kinetic model which accounts for all the phenomena of both low temperature, cool flame and ignition processes including induction periods has been completed. It is considered a major breakthrough in the kinetic understanding of flames and should open the way for the interpretation of a variety of researches from flame inhibition to knocking to flame-out (in jet engines) and to soot formation in flames. It is also capable of reproducing data on liquid phase oxidation and so should be applicable to lubricating oil studies and to droplet combustion.

#### Reports:

- No. 1-8 in previous edition.
9. Predicting Chemical Reactivity, by S. W. Benson, *Chemtech* 10, 121 (1980).

10. The Temperature Coefficient of the Rates in the System:  $\text{Cl} = + \text{CH}_3\text{CH}_3 + \text{HCl}$ ; Thermochemistry of the Methyl Radical, *Int J Chem Kin*, Shawn P. Heneghan et al.
11. A Possible Role for Metals in Stratospheric Chlorine Chemistry, by Edmond Murad et al.
12. The Prediction of Thermochemical and Kinetic Data for Gas Phase Reactions. Current Status, by Sidney W. Benson.

### 16124 CARS DIAGNOSTICS OF HIGH PRESSURE COMBUSTION

A. C. Eckbreth  
United Technologies Research Center  
SL: BRL SC: LCWSL

A room temperature series of experiments covering the pressure range of one to one hundred atmospheres has been completed. Concurrently, a theoretical model which predicts CARS spectra has been developed. The agreement between theory and experiment is quite good over the entire pressure range. A paper describing this work will be published in *Optics Communications*. Experimentally, high pressure, high temperature investigations of nitrogen gas over the range of one to 100 atmospheres and 300 to 1600K were carried out. The CARS signals became weak and erratic at higher temperatures and pressures because of extreme turbulence which produces large rapid density gradients. One result of this is the probable admixture of cooler nitrogen CARS signals with the hot nitrogen CARS signal; hence the spectra obtained are not easily interpreted. The magnitude and extent of the turbulent gradients will be reduced by the insertion of sapphire rods in the cell. Spectra taken recently of one atmosphere carbon dioxide at high temperature (up to 1500K) are in reasonable agreement with theoretically predicted spectra. A theoretical model for  $\text{CO}_2$  CARS spectra has been developed, and the initial comparison with one atmosphere spectra obtained on another show reasonably good agreement.

#### Reports:

1. Pressure-Induced Narrowing of the CARS Spectrum of  $\text{N}_2$ , by Robert J. Hall et al, *Opt Commun* 35, 69 (1980). AD A099 542
2. New Directions in CARS Diagnostics for Combustion, by John A. Shirley et al, *AIAA-80-1542*.
3. Combustion Diagnosis by Coherent Anti-Stokes Raman Spectroscopy (CARS), by Robert J. Hall et al, *Opt Eng*.
4. Spectroscopic Investigations of CARS for Combustion Applications, by Alan C. Eckbreth et al, *Adv in Laser Spectroscopy*.

### 16665 METAL SURFACE CORROSION STUDIES USING RAMAN AND SIMS PROBES

Robert M. Hexter  
University of Minnesota  
SL: LCWSL SC: MERADCOM, BWL

The vibrational frequencies of halogens adsorbed on Ag(100) have been calculated. The frequency of a single adsorbed atom vibration perpendicular to the surface is obtained from  $\text{AgX}$  ( $\text{X} = \text{F}, \text{Cl}, \text{Br}, \text{I}$ ) crystal frequencies. Interaction between dipoles consisting of the effective charge on the adsorbed atom and its induced image charge produces a coverage dependent frequency. The effective charge on the adsorbed atom is assumed to be  $1.0 e$ , while the electronic polarizability of the real-plus-image atom system is assumed to be twice the electronic polarizability of  $\text{X}^-$ . At full coverage, the calculated frequencies for the  $k = 0$  modes are 437, 241, 150, and  $113 \text{ cm}^{-1}$  respectively for  $\text{X} = \text{F}, \text{Cl}, \text{Br}, \text{I}$ , which compare well with experimental values, and these frequencies are assigned to external vibrational modes of halogen atoms adsorbed on silver.

#### Reports:

- No. 1-2 in previous edition.  
3. Vibrational Frequencies of Halogen Atoms Adsorbed on Silver Metal Surfaces, by Henry Nichols et al, *J Chem Phys*.

### 16780 STUDIES PERTAINING TO AEROSOL FORMATION: NUCLEATION PHENOMENA, CHARGING MECHANISMS AND GROWTH

Albert Welford Castleman, Jr.  
University of Colorado  
SL: CSL SC: ASL, HDL

As a continuing effort to elucidate the validity of conventional theories of aerosol formation via nucleation processes, additional comparisons have been made of our experimental ion clustering data with the liquid-drop formulation termed "the Thompson Equation." Work in the laboratory has established the fact that the Thompson Equation fails for many substances due to its inability to describe the ordering associated with small cluster development. This is manifested in its inability to properly account for the entropy contribution to

the energy barrier to nucleation. Comparison of theory and experiment has revealed that an upward adjustment of the surface tension for ion-containing droplets with decreasing droplet radius gives a somewhat better agreement between entropy and the appropriate derivatives of the Thompson Equation. While steady state nucleation formulations are useful approximations, a kinetic model will ultimately be required to give a proper description of the kinetics of phase transformations. In this regard, studies are being made of clustering kinetics. Experiments to measure the changing properties of clusters in order to follow the transition from the gaseous to the condensed state have advanced considerably. In particular, the applicability of a newly developed DEADHOT apparatus (deflection of electrically aligned dipoles by hexopole orientation technique) for studying clusters was established. Work has been completed for studies of ethyl alcohol, methyl alcohol, water and ammonia. In each case the dimers and the mixed dimers have been shown to have substantial dipole moments.

#### Reports:

- No. 1-5 in previous editions.  
6. Comment of Apparent Differences of Appearance Potentials Obtained by Photoionization and by Electron Impact Ionization of Rare Gas Dimers, by T. D. Mark et al, *J Chem Phys*.  
7. Studies of van der Waals Molecules by Electron Impact Ionization: Appearance Potentials of  $\text{Kr}_2$ ,  $(\text{CO}_2)_2$ , and Ammonia Clusters, by T. D. Mark et al *J Chem Phys*.  
8. Studies of the Structure and Bonding of Inorganic Clusters, by A. W. Castleman, Jr. et al, *J Surface Sci*.  
9. Experimental Studies of Ion Clustering: Relationship to Aerosol Formation Processes and Some Atmospheric Implications, by A. W. Castleman, Jr. *J Aerosol Sci*.  
10. Studies of Small Clusters: Relationship to Understanding Nucleation and Surfaces, by A. W. Castleman, Jr. et al, *J Vac Sci Tech*.  
11. Ion Association Processes and Ion Clustering: Elucidating Transitions from the Gaseous to the Condensed Phase, by A. W. Castleman, Jr. et al *J Radiation Phys and Chem*.

### 16856 LAGRANGE GAGE STUDIES OF NONIDEAL EXPLOSIVES CONTAINING $\text{NH}_4\text{NO}_3$

Michael Cowperthwaite  
J. Thomas Rosenberg  
SRI International  
SL: LCWSL SC: BRL

The overall objective of this research is to obtain a basic understanding of the behavior of ammonium

nitrate (AN) in the planar detonation process of composite military explosives formulated from TNT, RDX, and AN. The primary study material is Amatex 20, which is nominally 40% (by weight) TNT, 20% RDX, and 40% AN. The approach is first to perform and analyze Lagrange gage experiments in the baseline Amatex 20 to characterize the hydrodynamics of the detonation process and to determine detonation parameters. Detonation calculations with various degrees of completion of AN reaction are next performed and compared with the experimental results to determine the proportion of AN that supports the detonation wave. The explosive and the detonation process are termed nonideal in this work when not all the AN reacts to support the detonation wave. The amount and particle size of the AN are then varied and the procedures are repeated to complete determination of the behavior of AN in the composite explosives. In the previous research phase, measurements were made of the hydrodynamics and detonation parameters of baseline Amatex 20 and of Composition B and procedures were formulated for performing detonation calculations in nonideal explosives. The objectives of the current theoretical and experimental tasks are respectively to perform detonation calculations for determining the extent of AN reaction in the study materials and to investigate an Amatex 20 having a mean AN particle size differing from 0.7  $\mu\text{m}$ .

**16950 TIME RESOLVED STUDY OF  
PRIMARY DECOMPOSITION  
PROCESSES FOR NITRO PARAFFINS  
AND NITRAMINES**

Edward R. Grant  
Cornell University

SI: LCWSL, MICOM, BRL SC: BRL

Work has been completed on the multiphoton ionization (MPI) spectroscopy and dynamics of  $\text{NO}_2$ . From experiments carried out at high pressure in static cells, at low pressure in a quadrupole mass spectrometer ionization chamber, and in a molecular beam with ion mass analysis, an interesting and complex picture has been developed of the frequency dependence of competing ionization pathways in this molecule. At

wavelengths longer than 500 nm the main mechanism is direct ionization to  $\text{NO}_2^+$  via resonances at the level of the third photon in the  $3s\sigma$  Rydberg state of the parent  $\text{NO}_2$ . At wavelengths shorter than 500 nm, predissociation at the two photon level predominates. This yields neutral NO which goes on to ionize in the still present laser field. The qualitative nature of the MPI spectrum of this latter process changes as wavelength is tuned to the blue. In the near blue, around 490 nm, the spectrum is complex, indicating the formation of  $\text{O}(^1\text{P})$  and hot NO. At shorter wavelengths the spectrum simplifies indicating that predissociation changes to favor  $\text{O}(^1\text{D})$  and cold NO. One of the most important conclusions of this work is that to unambiguously detect primary  $\text{NO}_2$  from nitro compound decomposition one must use probe wavelengths longer than 500 nm. Recently the MPI probe technique has been applied to the successful detection of  $\text{NO}_2$  photoproduct in the multiphoton dissociation of  $\text{CH}_3\text{NO}_2$  in a molecular beam. Preliminary analysis indicates that the  $\text{NO}_2$  so produced is rotationally hot.

*Reports:*

1. Resonant Multiphoton Ionization Detection of the  $\text{NO}_2$  Fragment from Infrared Multiphoton Dissociation of  $\text{CH}_3\text{NO}_2$ , by Bennett H. Rockney et al, *Chem Phys Lett*
2. Two Photon Resonant Multiphoton Ionization Spectroscopy in the Ultraviolet: A New Rydberg System in  $\text{Br}_2$ , by Richard J. S. Morrison et al, *Chem Phys Lett*

**17369 CHEMISTRY OF ENERGETIC  
FRAGMENTS: HYPOVALENT  
RADICALS**

Richard McDonald  
Donald Setser  
Kansas State University  
SI: LCWSL SC: BRL

The flowing afterglow technique will be utilized in the synthesis of hypovalent radicals and in determining their chemistry. A variety of carbene, nitrene, and carbyne radical anions and cations, will be synthesized by the flowing afterglow (FA) technique. The ion-molecule reactions of these species with neutral reactants will be investigated and rate constants and product distributions determined. An attempt will also be made to develop the laser photodetachment technique and apply it to hypovalent radical anions.

**17416 LASER PROBES OF PROPELLANT COMBUSTION CHEMISTRY**

David R. Crosley  
SRI International  
SL: BRL, MERADCOM

The research objective is to investigate gas-phase flames pertinent to the burning of propellants, particularly the nitramines. Laser probe techniques such as laser-excited fluorescence on NH, NH<sub>2</sub>, CN and OH in CH<sub>2</sub>O/N<sub>2</sub>O flames will be utilized in concert with computer models of flame chemistry. Optical saturation methods will be extended, reaction rates will be measured in flowing systems, and rate constant estimation methods will be developed.

*Reports:*

1. Collisional Effects on Laser-Induced Fluorescence Flame Measurements, by David R. Crosley, *Opt Eng.*

**17623 QUANTUM CHEMICAL INVESTIGATIONS OF ENERGETIC MATERIALS**

Joyce J. Kaufman  
Johns Hopkins University  
SL: LCWSL, BRL SC: AMMRC

Theoretical investigations will be made on energetic compounds directed toward studying the decomposition of energetic molecules and their reaction pathways. Ab initio quantum chemical calculations will be conducted using MODPOT/VRDDO programs with the MERGE option.

**17710 FREE RADICAL DETECTION AND DYNAMICS IN PROPELLANT IGNITION AND COMBUSTION SYSTEMS**

John Stephenson  
National Bureau of Standards  
SL: BRL SC: LCWSL, BRL

The objective of the research is to investigate energy transfer processes and decomposition mechanisms of energetic molecules and to explore the effects of

additives on reaction rates and energy content on radical fragments and the effects of laser excitation on energetic species. Infrared multiphoton and ultraviolet photolysis techniques will be utilized to produce free radicals and ions which will be investigated using matrix isolation techniques which will be coupled to a variety of laser diagnostic methods.

**17967 FLOW REACTOR STUDIES OVER WIDE TEMPERATURE & PRESSURE RANGES OF ATOM & RADICAL REACTION RATES IMPORTANT IN IGNITION & COMBUSTION**

Frederick Kaufman  
University of Pittsburgh  
SL: BRL SC: LCWSL, MICOM

The objective of the research is to investigate elementary reaction rates important in combustion and ignition processes including the study of  $H+O_2 \rightarrow HO_2+M$ . Detailed rate measurements will be performed including product channel analysis of several two-body and three-body processes. A discharge-flow apparatus with a movable injector and fluorescence detection cell will be used to study reaction rates up to 1000K and pressures to about 100 torr. Flow parameters will be measured to establish laminar flow and the flow system will be characterized by mathematical and computer analysis.

**18039 THE DYNAMICS OF VIBRATIONALLY EXCITED MOLECULES**

C. Bradley Moore  
University of California at Berkeley  
SL: LCWSL SC: BRL

The objective of the research is to study the dynamics of vibrationally excited molecules and in particular to investigate the dynamics of chemical reaction and the competition between chemical reaction and vibration energy transfers in liquids and to continue the study of vibrational energy transfer in solid matrices. Product fragments, their kinetics of formation and energy distribution will



be investigated using improved tunable laser instrumentation and molecular beam techniques.

**18139 LASER AUGMENTED REACTION RATES - APPLICATION TO INTERCONVERSION AND OXIDATION OF THE BORANES**

Simon H. Bauer  
Cornell University  
SL: MICOM SC: MICOM, ARRADCOM

The research objective is to study processes leading to a diborane generation and study oxidation of borane adducts. The preparation of dimethoxyborane using a microwave discharge-flow reactor and its subsequent decomposition into diborane will be investigated. Oxidations of borane adducts with  $O(^3P)$  will be investigated.

**18172 PREDICTION OF THERMAL RATE CONSTANTS FOR COMBUSTION**

Bruce C. Garrett  
Michael J. Redmon  
Chemical Dynamics Corporation  
SL: BRL SC: MICOM

The objective of the research is develop and apply theoretical methods based upon generalized transition state theory to predict thermal rate constants for chemical reactions in combustion processes. Generalized transition state theory will be utilized in predicting thermal rate constants and will be extended to large systems using the harmonic approximation.

**18200 VAPOR EXPLOSIONS IN SUPERHEATED LIQUIDS**

G. Richard Fowles  
Washington State University  
SC: BRL

An experimental study will be made of the fundamental processes that occur during vapor explosions in superheated liquids. Particular emphasis will be placed on a test of detonation

theory, proposed as a possible mechanism for such explosions. A gas gun will be utilized in conjunction with an instrumented aluminum buffer target capable of nanosecond time resolution. Laser interferometry and electromagnetic gages will be used to monitor particle velocities.

**18271 ELECTRONICALLY EXCITED ALKALINE EARTH OXIDES: CHEMICAL PRODUCTION & COLLISIONAL ENERGY TRANSFER PROCESSES**

Paul J. Dagdigan  
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SC: LCWSL

The research objectives are to investigate the reactions of electronically excited alkaline earth atoms with various oxidants including the probing of the reactivity of individual spin-orbit states and to study inter-electronic energy transfer between molecular excited states. Optical-pumping state selection techniques will be used in the reactive studies and electrical discharges will be used to generate metastable electronically excited atoms. Theoretical techniques will be utilized in modeling the various kinetic processes.

**E. POLYMER CHEMISTRY**

**15466 MISCIBLE POLYMER BLENDS ON POLYCARBONATE AND A COPOLYESTER**

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SL: AMMRC, NLABS SC: MERADCOM, MICOM

Previous work indicated that melt processed blends of polycarbonate and copolyester developed a brilliant yellow coloration, not present in either pure component. It was theorized that color formation was probably the result of a complex formed between the residual titanium catalyst in the copolyester and reacted bisphenol A on the polycarbonate molecule, either present initially or generated as a result of transesterification reactions with the polyester. Recent experiments have shown

that the copolyester does contain 68 ppm titanium and that titanium catalysts added to phenol and bisphenol A form the yellow coloration found in the blend. In addition it has been determined that the transesterification and color development problems can be partially eliminated by the addition of small amounts of  $As_2O_3$  to the blend. Carbon dioxide sorption isotherms were measured for all blend compositions at 35°C, and these results were analyzed in the Henry's Law region to obtain the free energy of interaction, or chi parameter for the polycarbonate-copolyester system. Another measure of this parameter has been obtained from DSC measurements of the copolyester melting points in the pure state and in the blends. The chi interaction parameter is found to be negative by both measurements, and the magnitudes of the parameters agree to within 30% which suggests that the chi parameter changes slowly with temperature from 35°C to 270°C.

#### Reports:

1. Properties of Compatible Blends of Polycarbonate and a Copolyester, by P. Masi et al. *VIII Intl Congress on Rheology*.
2. Gas Sorption and Transport in a Copolyester and its Blends with Polycarbonate, by P. Masi et al. *J Polymer Sci*.
3. Mechanical Properties of Miscible Polycarbonate-Copolyester Blends, by E. A. Joseph et al. *Polymer*.

### 15601 SHEAR-INDUCED CHANGES IN POLYMER COMPOSITION AND MOLECULAR WEIGHT

Roger S. Porter  
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SI: MERADCOM SC: AMMRC, ETDL

The flow behavior at 25°C for decalin solutions of polystyrenes (PS) of molecular weights up to  $7 \times 10^6$  and polyisobutenes (PIB) of molecular weights up to  $3 \times 10^6$  was examined over a wide range of shear rate up to about  $10^5 \text{ sec}^{-1}$  for several polymer concentrations. Gel permeation chromatography was used to determine polymer molecular weight, its distribution, and possible change on shearing. Shear degradation was observed for polymers at high values of concentration, molecular weight, and shear stress. Poly(vinyl acetate) (PVAc) of Mw 750,000,

Mw/Mn 3.85 and Bn (the number of long branches per molecule) 2.2 was subjected to chain scission by mechanical (high-speed stirring) as well as chemical (saponification and reacetylation) methods to investigate the effect of shear on the branching distribution. The extent of long branching was measured by gel permeation chromatography on-line with low-angle laser light scattering photometry. It was concluded that (i) the branches through the acetate group are long and are ruptured preferentially on shearing, (ii) the branches through the  $\alpha$ - and  $\beta$ -carbons are not broken on shearing, (iii) the extent of long branching through the acetate group is about 67% of total branching, (iv) on shearing, 80% of the decrease in molecular weight is due to rupture of the long branches through the acetate group. The remaining 20% of the decrease in molecular weight results from the main chain scission; and (v) the poly(vinyl alcohol) derived from branched PVAc contains a smaller but nevertheless significant amount of branching.

#### Reports:

No. 1-2 in previous edition.

### 15770 THE EXAMINATION OF SECONDARY RELAXATION PHENOMENA IN THE SOLID STATE BY MEANS OF EXCIMER FLUORESCENCE

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Stanford University  
SC: AMMRC, MERADCOM, CENCOMS

The majority of the recent efforts have been devoted to instrumentation development. The first area of equipment development involved the thermostated sample chamber into which are placed the thin films of the blends of polystyrene/poly(vinylmethylether). Although the existing film holder was quite adequate for isothermal experiments, its relatively large size made it difficult to obtain fast heating or cooling rates or rapid step changes in temperature. A new film holder with considerably smaller thermal mass was designed and fabricated for this purpose. The second area of instrumentation development involved the polychromator detection system. This system is based on a one meter SPEX monochromator/spectrograph with provision for multiple detection channels. The initial

experiments were performed using two channels, one each for the excimer and monomer emission intensities. Since scattered light has proven to be somewhat variable in the blends, it was necessary to add an additional detection channel to measure it directly. This permits appropriate corrections to be made to the monomer emission signal. Another detection channel has been added to measure the low energy side of the excimer band to determine whether there is any band shift with temperature.

#### Reports:

1. The Study of Conformational Statistics in Rubbery Blends of PS/PVME by Excimer Fluorescence, by Richard Gelles et al.

### 15795 DYNAMIC ORIENTATION STUDIES OF POLYMERS

R. S. Stein

University of Massachusetts

SL: AMMRC SC: MERADCOM, CENCOMS

Measurements of dynamic small x-ray scattering at Oak Ridge National Laboratory have been initiated. Preliminary measurements on low density polyethylene have indicated the feasibility of measuring the angular dependence of the amplitude and phase angle of changes in scattered intensity, although increases in precision are desirable. These measurements characterize the dynamics of lamellar orientation within spherulites. The relative importance of form and deformation birefringence to the negative birefringence found for high density polyethylene at low temperatures, high frequencies or short times (in relaxation) has been studied by examining the effect of swelling of birefringence. There are two effects, an irreversible one associated with a loss of orientation associated with relaxation caused by the plasticization by the solvent, and a reversible one believed related to the effect of the solvent on the refractor index of the swollen amorphous phase which affects the form birefringence. The reversible effect is only a small part of the total birefringence. Consequently, it is believed that this form birefringence contribution is not sufficiently large to account for the negative total birefringence and it is now believed that the negative value is associated with a deformation birefringence contribution. However, it is necessary to establish what the effect of the solvent is on

crystal orientation rates. For this purpose, dynamic x-ray diffraction measurements on swollen samples are planned.

#### Reports:

- No. 1-8 in previous editions.
9. Longitudinal Acoustical Mode in Linear Aliphatic Polyesters, by Y. K. Wang et al, *J Polymer Sci-Phys*.
10. Morphological Studies of Poly(Vinylidene Fluoride) and its Blends with Poly(Methyl Methacrylate), by Bruce S. Morra et al.
11. Melting Studies of Poly(Vinylidene Fluoride) and its Blends with Poly(Methyl Methacrylate), by Bruce S. Morra et al.

### 16086 INVESTIGATION OF INDUCTION PERIODS IN PHOTOOXIDATIONS OF STABILIZED POLYPROPYLENES

Frank R. Mayo

SRI International

SL: AMMRC SC: CENCOMS, MERADCOM

The object of this research is to predict service lives of stabilized polypropylene (PP) films by extrapolation of the very slight reactions that occur during the initial exposures to light or heat. These slight changes during the induction periods (I.P.) are not measurable by conventional methods but two new and much more sensitive methods have been developed. One of these, applicable to both photo- and thermal oxidations, involves oxidation in synthetic air and following the changing O<sub>2</sub>/N<sub>2</sub> ratio by gas chromatography. Automation of the sampling and gc analysis is nearly complete. In photooxidations of Film I, containing both a phenolic inhibitor and a peroxide decomposer (di-tert-butyl thiodipropionate), the rate of oxygen absorption is slow and steady until the film has absorbed about half the oxygen ultimately absorbed at failure (brittleness); it then increases gradually. Film F, containing both a hindered piperidine and a phenolic inhibitor, gives a somewhat higher initial rate of oxidation, a slowing, and a sharp increase in rate when the oxygen supply is replenished. The initial absorption of oxygen corresponds approximately to that required to convert the amine to a nitroxyl radical, the effective stabilizer. The dependence of rate on oxygen concentration apparently has not been reported previously and has interesting implications for aging and stabilization with hindered piperidines. Radiation reduces or

eliminates the induction periods but not in a way that will assist in prediction of service life. In thermal oxidations at 150°C, several stabilized films show no measurable oxidation until there is a sudden onset of rapid oxidation and quick failure.  $\gamma$ -Radiation (0.5 megarad) greatly reduces this IP and 2 megarad results in immediate rapid oxidation. These results indicate that efforts to measure oxygen absorption during induction periods at 150 degrees are unpromising but the second new method, thermogravimetric analysis at 176 degrees, may be useful. This method measures the stabilizer surviving in oxidations at lower temperature.

#### 16144 FUNDAMENTAL INVESTIGATIONS IN POLYMER CHEMISTRY

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David R. Squire  
North Carolina State University  
SC: NLABS

The work on the influence of monomer and solvent effects has resulted in the submission of three manuscripts for publication. The findings indicate that there is little or no solvation of the propagating species by the monomer. When even small quantities of methylene chloride are present the results indicate that these molecules solvate the free ions and not the monomer. Further, the results indicate that in certain circumstances there could be a self-solvation of the propagating cations by their own chains. Work on a comprehensive paper entitled "The Effect of Radiation on Some Polyphosphazenes" is nearing completion. The broad and sometimes bimodal nature of the molecular weight distributions of these polymers has necessitated a more extensive treatment of the data than the normally used sol gel analysis techniques.

##### Reports:

No. 1-2 in previous edition.

3. The Effect of Solvents on the Radiation-Induced Polymerization of Ethyl and Isopropyl Vinyl Ethers, by W. C. Hsieh et al.
4. The Radiation-Induced Cationic Polymerization of p-Methoxystyrene in Bulk and in Methylene Chloride Solution, by A. Deffieux et al, *Polymer Bull.*
5. The Radiation-Induced Cationic Polymerization of Vinyl Ethers in Solution, Part I. Influence of Monomer and Solvent Concentration on the Rate of Polymerization of Ethyl Vinyl Ether in Benzene, by A. Deffieux et al.

6. The Radiation-Induced Cationic Polymerization of Vinyl Ethers in Solution, Part II. Polymerization of Ethyl Vinyl Ether in Methylene Dichloride Solution, by A. Deffieux et al.
7. The Radiation-Induced Cationic Polymerization of Vinyl Ethers in Solution, Part III. The Influence of Monomer and Solvent Concentrations on the Rate of Polymerization of Ethyl Vinyl Ether in Ether Solvents, by W. C. Hsieh et al.

#### 16232 FABRICABLE POLYMERS WITH EXCELLENT HEAT, OXIDATIVE AND HYDROLYTIC STABILITY

C. S. Marvel  
University of Arizona  
SL: AMMRC SC: NLABS, CSL, MERADCOM

Work on the preparation of oligomeric benzimidazoles with reactive end groups was terminated due to the difficulty in obtaining oligomers in satisfactory yields and of satisfactory quantity. About the time this project was well underway, Dr. White and Dr. Steg of the Chemical Engineering Department had found that a ether-keto-sulfone resin (Swedo and Marvel, *Polymer Letters*, 15, 683 (1977)) was proving to be an exciting resin for laminates. Additional polymers of this type were prepared. The original sample contained 5 mole % of the isophthaloyl chloride replaced by the biphenylene acid chloride. That choice was made arbitrarily without any reason for selection. Later it was decided that polymers would be made with less biphenylene unit and more biphenylene unit, say, 2 1/2 mole % to 10% in order to pinpoint the exact amount that would be best suitable for laminate formations. A paper is being prepared which describes the qualities of the new laminating powder. Other work has been concerned with preparing samples of the aromatic poly-ether-ketones and aromatic poly-ether-keto-sulfones with biphenylene units for crosslinking.

#### 16357 THE STRUCTURE OF POLYURETHANE ELASTOMERS

John Blackwell  
Case Western Research University  
SL: AMMRC SC: MICOM, MERADCOM

In previous work the structure of methanol-capped MDI was determined. This structure has been used



to propose a model for the chain conformation and packing of MDI-butandiol hard segments. The structure of butandiol capped MDI has now been determined but not completely analyzed. X-ray patterns of MDI-butandiol-poly(tetraethylene adipate) systems containing 50% hard segments, and in which the soft segments are amorphous have been obtained. This work has now been expanded to other polyurethanes, with preparations using MDI with propandiol glycol and ethylene glycol as the chain extender. Based on the model compound and polymer x-ray work, minimum energy conformational analysis is being used to predict the most likely chain conformations for MDI with butandiol, propandiol, and ethylene glycol as the chain extenders. Initial work has concentrated on the butandiol structure.

#### Reports.

No. 1-2 in previous edition.

### 16389 MODEL NETWORK POLYMERS

Christopher W. Macosko  
University of Minnesota  
SL: AMMRC SC: ETDI.

**Crosslinking of Long Chains:** Poly (dimethyl-co-methylvinyl-siloxane) was crosslinked by reacting with a dihydrosilane ( $\text{HSi}(\text{CH}_3)_2\text{OSi}(\phi)_2\text{O}(\text{CH}_3)_2\text{SiH}$ ) using a platinum catalyst. This generates randomly linked long chains as in typical sulfur or peroxide crosslinking but there is no problem of chain scission or chain polymerization. The number of linkages can be determined from conversion of the SiH and using branching theory. The results for modulus and sol fraction agree well with theory if entanglements are included and the general results of previous endlinked networks. Large strain results do not fit the Mooney Rivlin relation but do agree with Flory's new theory if a chain entanglement term is added. **Endlinked Networks:** The work of Valles is being extended to a wider range of starting molecular weights. Both broad and narrow distribution vinyl-poly(dimethylsiloxane) chains are reacted with 3 and 4 functional hydrosilane crosslinkers. It is necessary to use excess SiH to completely end link all the siloxane. This indicates a small side reaction which investigated further. **Randomly Branched Chains:** Using the endlinking system above with

enough excess vinyl to prevent gelation, randomly branched chains can be made. Their molecular weight is being measured by light scattering and their rheology.

#### Reports:

1. Equilibrium Modulus of Model Poly(Dimethylsiloxane) Networks, by M. Gottlieb et al, *Macromol*
2. Modulus of Three and Four Functional Poly(Dimethylsiloxane) Networks, by Christopher W. Macosko et al.
3. Substitution Effects in Property Relations for Stepwise Polyfunctional Polymerization, by Douglas R. Miller et al, *Macromol* 13, 1063 (1980). AD A095 674.
4. Stress Strain Behavior of Randomly Crosslinked Poly-(Dimethylsiloxane) Networks, by M. Gottlieb et al, *Polymer Sci.*

### 16426 CONFORMATION AND DYNAMICS OF POLYMERS IN SOLUTION

Ben Chu  
State University of New York at Stony Brook  
SL: AMMRC SC: MERADCOM

A new method has been devised for studying polymerization kinetics based on Raman scattering and the possibility of using Raman scattering, angular distribution of time-averaged scattered intensity and photon correlation spectroscopy has been explored to examine the thermal polymerization of styrene and of hexachlorocyclo-triphosphazene. In the applications, use was made of a double monochromator and an optical system capable of making virtually simultaneous detections of Raman scattered intensity and Rayleigh scattering including photon correlation at three different scattering angles, as well as an estimate of the refractive index of the solution. The intensities of Raman lines have been calibrated using classical analytical techniques and the polymerization kinetics compared with literature values. The method has been illustrated to be fairly general, easy to use and to automate and insensitive to most spurious phenomena. Therefore, there is potential in applying the method to industrial processes.

#### Reports:

1. Osmotic Compressibility and Linewidth of (NBS-705) Polystyrene in Methyl Acetate, by K. Abbey et al.
2. Static and Dynamic Properties of Polymer solutions, by B. Chu.
3. Mapping of Static and Dynamical Properties Polystyrene (NBS 705 Standard) in Methylacetate, by B. Chu et al.

4. Static and Dynamical Properties of Polystyrene in Carbon Tetrachloride. 1. Characteristic Frequency in the Dilute, Intermediate, and Semidilute Regions, by Y. -H. Lin et al, *Macromol* 14, 385 (1981).
5. Static and Dynamical Properties of Polystyrene in Carbon Tetrachloride. 2. Osmotic Pressure and Radius of Gyration in the Dilute, Intermediate, and Semidilute Regions, by B. Chu et al, *Macromol* 14, 392 (1981).
6. Study of Thermal Polymerization of Styrene by Raman Scattering, by B. Chu et al *Macromol* 14, 395 (1981).

### 16577 MECHANISMS OF CHAR FORMATION DURING POLYMER FLAMMABILITY

Eli M. Pearce  
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SL: NLABS SC: AMMRC

The structure study of novolac oligomers with various substituents was completed using NMR spectroscopy. The results indicated the presence of the methylene bridges formed during the condensation of phenol and formaldehyde in novolac oligomers resulted in three different forms assigned as para-para<sup>1</sup>, ortho-ortho<sup>1</sup>, ortho-para depending upon the positions of hydroxyl groups on the adjacent aromatic rings relative in the methylene bridge. The chemical shifts of the methylene protons for nonsubstituted and meta-substituted phenol-formaldehyde oligomers depend on the linkages and electronegativities of the substituents. All assignments of the chemical shifts in NMR spectra were completed. All structures of oligomers and cured phenolic polymers were completed using FT-IR spectroscopy. These results indicated the same chemical structures of novolac and resol cured halogenated phenolics which were cross-linked via methylene bridges having particularly the same structures, with the small additional amounts of ether linkages which were splitting to form the corresponding aldehydes. The same structures of these polymers have good correlation with the new thermooxidative data and thermogravimetry, elemental analysis and Oxygen Index (OI). The phenolic polymers were cured with p-terephthaloyl chloride to form ester bridges. The presence of such bridges and the absence of available hydroxyls were definitely recorded in FT-IR spectra. The OI of these halogenated phenolic polymers decreased to 34-46, compared to 74-75 for the novolac and resol cured polymers. Evaluation was made of the effect

of varying ratios of meta-halogen-substituted phenols and phenol with different crosslinking agents on the thermal behavior of their copolymers. The results surprisingly indicated no increase of OI for copolymers which were synthesized by the novolac route, whereas good increases in OI were obtained for the copolymers synthesized by the resol approach.

#### Reports:

- No. 1 in previous edition.
2. Some Structure-Property Relationships in Polymer - Flammability: Studies of Phenolic-Derived Polymers, by J. Zaks et al, *J Polymer Chem.*

### 16662 SIMULTANEOUS REACTING RELAXING POLYMER SYSTEMS

John J. Aklonis  
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SL: AMMRC SC: MERADCOM, NLABS

The investigation of the behavior of simultaneously chemically reacting and physically relaxing networks is continuing. In addition to furthering the investigation based on the photochemistry of disulfides, a study using photodimerization of anthracene is in progress. The properties of glasses have been studied as they vary during annealing. The annealing process causes changes in glasses which are mathematically similar to those induced via chemical reactions in relaxing elastomers. It was previously determined that time cross-link density reduction is applicable to the creep behavior of crosslinked polyurethane networks. This work was done by photochemically varying network crosslink density through the photolysis of disulfides. This original work was all carried out on networks with tri-functional crosslinks. Since quantitative aspects of the reduction were different from what had previously been reported in the literature, it was decided to investigate the possibility that networks with crosslinks of various functionality might act differently from one another. Thus, tetrafunctional crosslinking agents were employed in a series of experiments. The results clearly showed that functionality of the crosslinking agent is not an important variable in determining behavior in this domain. Results in the area of simultaneously relaxing and reacting polymer networks further the development of a technique which can be employed to predict long

term properties of elastomers under real service conditions. In addition, fundamental information concerning the effect of specific changes in network topology on the viscoelastic properties of elastomers in the terminal relaxation zone have been published and are forthcoming. A new and successful theory of glass transition has been completed. The results are particularly interesting since they indicate that the motion of molecules in polymeric materials may be very strongly influenced by thermal history of the sample. The results infer that diffusional motion of small molecules in glasses might be modified by factors greater than 1000, merely by subjecting the material to an appropriate thermal history.

#### Reports:

- No. 1 in previous edition.
- 2. Kinetic Treatments of Glass Transition Phenomena and Viscoelastic Properties of Glasses, by J. J. Aklonis, *Polymer Sci and Eng.*
- 3. Relaxation of Damped Debye Lattices: Polymethylmethacrylate, by Kang Chi Lin et al, *Polymer Sci and Eng.*
- 4. Evaluation of the Stress-Relaxation Modulus for Materials with Rapid Relaxation Rates, by K.S.C. Lin et al, *J Appl Phys* 51, 5125 (1980). AD A093 547
- 5. Isothermal Viscosity Variation in Glasses Measured by Fluorescence Depolarization, by Been-Jon King-Wood et al.

### 16728 CHEMICAL DESIGN OF ELASTOMERIC BEHAVIOR

Jack L. Koenig  
Case Western Reserve University  
SI: AMMRC SC: MERADCOM

The molecular structures of the coupling agents  $\gamma$ -aminopropyltriethoxysilane (APS), N-methylaminopropyltrimethoxysilane (MAPS), and N-2-aminoethyl-3-aminopropyltrimethoxysilane (AAPS) have been studied in aqueous solutions and as solids by Fourier transform infrared spectroscopy (FT-IR). The structure of the silanes condensed as bulk polymers and adsorbed on glass surfaces have also been investigated by high resolution solid state carbon-13 nuclear magnetic resonance spectroscopy. The resonance peaks of low-cured and high-cured polyaminopropylsiloxanes show different chemical shifts. The results are consistent with a theory of chemical shifts being induced by electric field effects of the C<sub>2</sub> carbon for

different aminosilane isomers. Interfacial chemical bonds are formed at the interface of the aminosilane and the nadic methyl anhydride cured epoxy resin in fiber reinforced composites at elevated temperatures. In comparing the relative reactivities of the two coupling agents toward epoxy resin, the secondary aminosilane has a higher reactivity than the primary aminosilane. A fiber pullout test has been developed to provide the shear strength of the fiber/matrix interface of the silane-treated fiber composites. The correlation of the interfacial strength and the molecular structure at the interface of composite is expected. The results of this mechanical test can be used to improve the mechanical strength and hydrothermal stability of the composites.

#### Reports:

- 1. Spectroscopic Characterization of the Matrix-Silane Coupling Agent Interface in Fiber Reinforced Composites, by Chwan-hwa Chiang et al, *J Polymer Sci: Phys.*
- 2. Comparison of Primary and Secondary Aminosilane Coupling Agents in Anhydride-Cured Epoxy Fiberglass Composites, by Chwan-hwa et al, *J Polymer Sci: Phys.*

### 16746 CROSS-LINKING REACTIONS OF THERMALLY STABLE, HIGH PERFORMANCE POLYMERS

John K. Stille  
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SI: AMMRC SC: LCWSL, NLABS, MERADCOM, HDL

Cross-linking agents containing two biphenylene units linked by thermally stable aromatic units were synthesized such that the aromatic units resembled certain thermally stable polymers the bisbiphenylenes were intended to cure. Thus, bisbiphenylenes linked by quinoline, isophthalamide, pyromellitimide, and diphenyl ether groups were used to cross-link polyquinoline, polyaramide, polypyromellitimide, poly(ether sulfone), and poly(phenylene ether) samples. Films from solutions containing polymer, cross-linking agent, and, in some examples, a transition-metal catalyst were plasticized, resulting in lower glass transition temperatures and dynamic storage moduli than film samples devoid of cross-linking agent and catalyst. A series of polyquinolines with one or two "cardo" fluorenyl and/or anthronyl-10

units in the polymer main chain was synthesized from the appropriate "cardo" bis oaminobenzophenones or bis acetyls. All the polyquinolines show good solubility in chlorinated hydrocarbons and excellent thermal stability. The glass transition temperatures ( $T_g$ ) of these polyquinolines ranged from 300-420°C. The self condensations of 5-acetyl-2-aminobenzophenone, 4-amino-4'-acetyl-3-benzoylbiphenyl and copolymerization of these monomers were carried out in a *m*-cresol/di-*m*-cresyl phosphate medium to afford rigid-rod polymers that possessed high  $T_g$ 's (330-415°C), limited solubilities, and retention of a relatively high percentage of mechanical properties above  $T_g$ .

#### Reports:

- No. 1 in previous edition.
- Dilute Solution Parameters for a Fluorene Containing "Cardo" Polyquinoline, by S. M. Padaki et al, *Macromol.*
- Polyquinolines, by J. K. Stille, *Macromol.*
- Synthesis and Properties of Rigid-Rod Polyquinolines, by P. D. Sybert et al, *Macromol.*
- Polyquinolines Containing Fluorene and Anthrone "Cardo" Units: Synthesis and Properties, by J. K. Stille et al, *Macromol.*

### 16796 EPITAXIAL POLYMERIZATION AS A MOLECULAR ENGINEERING TECHNIQUE

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Case Western Reserve University

SL: AMMRC, FCSCWSL SC: MERADCOM, AMMRC

Study of hexachlorocyclotriphosphazene ( $N_3P_3Cl_6$ ) has continued. This compound crystallizes epitaxially upon sublimation at  $10^{-5}$  torr and 100°C. The substrate used is a freshly cut NaCl crystal. Study of the morphology and structure of the epitaxial crystals of the  $N_3P_3Cl_6$  and the resulting polymer continues in order to verify preliminary findings. In addition, another compound, hexamethylcyclotriphosphazene  $N_3P_3(CH_3)_6$  has been prepared. The methyl group is expected to render this compound stable against hydrolysis (as opposed to the chlorine compound) and the epitaxial polymerization should lead to a hydrolytically stable polymer. Both the chlorine and methyltriphosphazenes are epitaxially

crystallized by sublimation in an apparatus devised especially for this technique. Investigation of the new crystal structures of (SN)<sub>n</sub> will be continued.

### 16961 INTERURETHANE HYDROGEN BONDING IN THERMOPLASTIC POLYURETHANE ELASTOMERS

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SL: MICOM, AMMRC SC: MERADCOM, HDL

A series of segmented polyurethanes based on a hydroxyl terminated polybutadiene soft segment (HTPBD) has been prepared with varying hard segment content between 20 and 60 weight percent. These materials are linear and amorphous and have no potential for hydrogen bonding between the hard and soft segments. The existence of two-phase morphology was deduced from dynamic mechanical behavior and thermal analysis. Both techniques showed a soft segment glass transition temperature,  $T_g$ s, at -56°C and hard segment transitions between 20 and 100°C, depending on the urethane content. The low value of  $T_g$ s, only 8 degrees higher than the  $T_g$  of free HTPBD and independent of hard segment concentration indicated nearly complete phase segregation. Depending on the nature of the continuous and dispersed phases, the urethanes behaved as elastomers below 40 weight percent hard segment or as glasslike materials at higher hard segment contents. The effect of thermal history on transitions of the HTPBD-urethanes was also investigated and the results suggest that the absence of hydrogen bonding to the soft segment must account for the extraordinary insensitivity to thermal history in dynamic mechanical, thermal and stress-strain behavior. Comparisons were made with the more common polyurethanes containing polyether and polyester soft segments.

#### Reports:

- Structural and Mechanical Properties of Polybutadiene-Containing Polyurethanes, by C. M. Brunette et al, *Polymer Eng Sci.*
- Thermal and Mechanical Properties of Linear Segmented Polyurethanes with Butadiene Soft Segments, by C. M. Brunette et al, *Polymer Eng Sci.*



**17312 ELECTROHYDRODYNAMIC AND FLOW INSTABILITIES IN NEMATIC POLYMERS**

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Duke University  
SC: MERADCOM, AMMRC, NLABS

The objective of the research is to investigate alignment effects of surfaces and electric fields, electric field-induced transitions and electrohydrodynamic instabilities, and anisotropic viscosity and flow instabilities in polymeric mesophases. The role of conductivity versus charge injection in the development of instabilities will be investigated. Anisotropic viscosity will be investigated using the Gahwiller technique with a magnetic field. Optical and photographic techniques will be used to study polymer flow and in the characterization of the type of instability mode.

**17438 PHYSICAL PROPERTIES AND STRUCTURE OF ION-CONTAINING POLYMERS**

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McGill University  
SL: NLABS SC: MERADCOM, LEWIS

The research objective is the investigation of ion-containing polymers to include: structure-property relations in ionomers; structure of ionic aggregates; ion-hopping phenomena; and plasticized ionomer systems. Stress relaxation techniques, dynamic mechanical tests, and melt rheology studies will be carried out on a wide variety of well-characterized polymers to establish structure property relationships. Raman spectroscopy, FTIR, SANS, and a variety of spectroscopic techniques will be utilized to establish the structure of the ionic aggregates. Detailed kinetic studies will be carried out for the process of motion of an ion or ion pair from one aggregate to another. Preliminary studies of plasticized ionomer systems will be carried out. A study of the dynamic mechanical properties of quaternized styrene - 4 vinyl pyridine copolymers

has now been completed. Surprisingly, no extensive clustering seems to be present in these ionomers. A stress relaxation study still remains to be performed. A study of the synthesis of copolymers of ethyl acrylate (EA) and vinyl pyridine (VP) has now been completed, and the synthesis of large samples will commence in the near future. This study is of particular interest since it was shown that ionic interactions are extensive in quaternized butadiene copolymer of VP, while they are minimized in styrene copolymers. The EA system should represent an intermediate case. Under previous industrial sponsorship, an instrument was designed for the measurement of stress relaxation under water. The instrument was subsequently modified in such a way as to allow the measurement of stress relaxation during the passage of current through a membrane.

**17559 MATRIX ENDOR STUDIES OF RADICAL MATRIX INTERACTIONS IN POLYMERS AND ON SURFACES**

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University of Houston  
SL: ETDL

The research objective is to investigate radiation damage in lithographic polymers and the nature of radical-surface interactions. Radical-matrix interactions in polymers and surfaces will be studied by matrix ENDOR. A manuscript has been completed on the importance of nuclear spin diffusion as an angularly independent relaxation mechanism controlling the matrix ENDOR response. An analysis of gamma-irradiated lithographic polymers by electron spin resonance and ENDOR is in progress. A model of g-anisotropy averaging of peroxy radical spectra due to molecular motion has been extended to poly(tetrafluoroethylene).

**Reports:**

1. The Importance of Nuclear Spin Diffusion as an Angularly Independent Relaxation Mechanism Controlling the Matrix ENDOR Response of Radicals in Molecular Crystals, by Shulamith Schlick et al, *Chem Phys*.

# 17617 HIGH PERFORMANCE ELASTOMER AND OTHER MULTIPHASE ORGANIC COMPOSITES

J. E. McGrath  
G. L. Wilkes  
D. W. Dwight  
T. C. Ward  
D. G. Baird

Virginia Polytechnic Institute and State University  
SL: MERADCOM, AMMRC SC: AMMRC, LEWIS

An integrated, multidisciplinary effort will be made to obtain a detailed fundamental understanding of novel, high performance elastomers and multiphase polymeric composites. The study will emphasize copolymer synthesis and molecular characterization and encompass synthesis and molecular characterization and investigations of bulk mechanical behavior (stress, strain, impact, dynamic mechanical) dielectric relaxation and novel surface behavior. A systematic examination of morphological, surface energetic, rheological and mechanical properties of copolymers as hot melt and pressure sensitive adhesives will be carried out. Films, elastomers and metals will be laminated and post fracture and prefabrication examinations of the laminates will be made to correlate bond strength and failure fractography. Proper methodology for surface studies of these polymers will be developed and ESCA and SEM studies will be carried out. Finally, correlations between the rheological properties and processing conditions with the final structure-property relationships in thermotropic liquid crystalline materials will be developed. Rheological studies will be used to design molds and dies which will result in optimum orientation in the melt state. Morphological structure and physical properties will be determined and correlations developed.

# 17773 PERMEATION OF MIXED PENETRANTS THROUGH GLASSY POLYMER MEMBRANES

W. J. Koros  
North Carolina State University  
SL: NLABS, CSL SC: CSL

A program will be conducted to measure and analyze the permeation of mixed gases in highly

permselective glassy polymers. The program will be achieved by: (1) measurement of high pressure sorption and permeation of pure and mixed penetrants in kapton polyimide; (2) generalization of the dual mobility model for steady-state permeation of gas mixtures; and (3) investigation of possible deviations from simple dual mode permeation behavior.

# 17847 CHEMICAL PHYSICS: FILM PROTECTION OF POLYMERS

H. L. Frisch  
State University of New York at Albany  
SL: NLABS, CENCOMS, CSL SC: CSL

The novel concept of utilizing polymer substrates as chemically inert, sacrificial, self-regenerating films will be explored both experimentally and with theoretical models. Candidates for self-regenerating films will be established and their chemical and physical properties established, e.g. kinetics of film formation, film thickness and morphology, and permeability to CW models. A simple mathematical model for the system will be established and studied for verification. Fundamental chemical physics related to this area will be investigated.

# 18001 PHYSICAL CHEMISTRY OF HIGH POLYMERS - SURFACE AND RELATED PROPERTIES

Harry L. Frisch  
State University of New York  
at Albany  
SL: CENCOMS SC: CSL, MICOM

The physical chemistry of polymers will be explored in order to provide a better understanding of the surface properties, mass transport, and permeability. Monte Carlo simulations of dense polymer systems will be carried out to elucidate the static and dynamic properties of these systems as a function of degree of polymerization and density. The free volume dependence of highly crystalline polymers and highly filled polymers will be studied in regard to mechanical and transport properties.

## II BIOLOGICAL SCIENCES

### A. FOOD AND RATION RESEARCH

#### 14926 ROLE OF ANTIBIOTICS IN SPORULATION

Arnold Demain  
Massachusetts Institute of Technology  
SL: NLABS

Studies have continued on the effects of the peptide antibiotic, gramicidin S (GS) on its producer, *Bacillus brevis*. It should be recalled that the presence of the antibiotic leads to the delay or inhibition of the spore outgrowth process (upon exposure to favorable conditions for growth, the dormant *Bacillus* spore passes through transformations called germination and outgrowth before entering vegetative growth with the first cell division). GS appears to act from outside the spores to inhibit/delay their outgrowth. Exogenous GS reproduces the natural effect of the presence or absence of endogenous antibiotic in spore preparations. Also, a mixture of parent and mutant spores are delayed in outgrowth, as if the GS in the parental spore preparation could diffuse and affect mutant spores. Thirdly, the inactivation of GS by treatment of parental spores with the hydrolytic enzyme Nagase, results in a significant reduction in the delay of outgrowth for Nagase-treated spores versus untreated spores. Studies have indicated that whereas mutant spores germinate, outgrow and begin vegetative growth in a rather homogeneous fashion, parental spores behave in a heterogeneous manner. Parental spores darken but then the vast majority remain as dark spores while only a few rods finally appear and grow, leading to the observed increase in absorbance. This implies that GS inhibits spore outgrowth indefinitely except for an occasional spore which escapes the effect. In nature, GS may inhibit spore outgrowth indefinitely, until the spores are more dispersed and survival conditions more positive.

#### Reports:

No. 1 in previous edition.

#### 15671 INTERRELATIONSHIP BETWEEN BOUND WATER AND MICROBIAL STRESS AND SURVIVAL IN MODEL AND FOOD SYSTEMS

M. P. Steinberg  
Lloyd D. Witter  
University of Illinois at Urbana  
SL: NLABS

A study is being made to determine the energy with which water is bound to food constituents and mixtures. The thermodynamic approach was taken; this is based on sorption data over a wide range of water activity ( $A_w$ ) but at three temperatures and involves Arrhenious plots. The hypothesis to be tested is that the composite binding energy for a mixture is equal to an energy balance of the binding energies of the individual ingredients. Another study is underway to learn the mechanism by which *Staphylococcus aureus* is able to tolerate such low  $A_w$  environments. Mutants were isolated which were incapable of multiplication under high salt conditions. By comparing the physiological responses of the wild type *S. aureus* MF-31 to the mutants, it is hoped that those responses necessary for adaptation and survival will be more fully understood.

#### 15904 RAPID DETERMINATION OF MICROBIAL NUMBERS IN FOODS

James M. Jay  
Wayne State University  
SL: NLABS SC: LAIR

In a soon-to-be published paper, the manner in which numbers of bacteria can be estimated in fresh ground beef in 1 h by use of the *Limulus* amoebocyte lysate (LAL) test is presented. While the procedure is shown to be workable, the confidence limits for samples within the various sample count ranges are rather wide. It is, of course, desirable to have these fall within acceptable ranges. Efforts are being concentrated on ways in

which the confidence limits can be sharpened. Although better results are achieved by use of the viable counts of gram negative bacteria obtained by use of a recently developed culture medium, better improvements are being sought. These consist of direct determinations of the endotoxin content of meat-borne gram negative bacteria followed by the application of these values to LAL-determined values for endotoxin in meats to calculate the number of cells. Since meats may be expected to contain some quantity of endotoxin referable to nonviable cells, attempts are being made to determine whether or not such value is consistent enough from sample to sample to be used to correct the calculated value for viable cells.

#### Reports:

- No. 1-2 in previous edition.
3. Comparison of Direct Serial Dilution and Most-Probable-Number Methods for Determining Endotoxins in Meats by the *Limulus* Amoebocyte Lysate Test, by Julie A. Seiter et al *Appl and Environ Microbio* 40, 177 (1980).
4. Analysis of Food Products for Microorganisms or Their Products by Nonculture Methods, by James M. Jay, *Food Analysis: Principles and Techniques*, ed by J. R. Whitaker et al, 1981, Marcel Dekker.
5. Incidence of Yeasts in Fresh Ground Beef and Their Ratios to Bacteria, by James M. Jay et al, *J Food Sci* 46, 648 (1981). AD A099 656
6. Rapid Estimation of Microbial Numbers in Fresh Ground Beef by Use of the *Limulus* Test, by James M. Jay, *J Food Protection* 44, 275 (1981).

#### 16648 PURIFICATION AND PROPERTIES OF CLOSTRIDIUM PERFRINGENS SPORE LYTIC ENZYMES

Ronald G. Labbe  
University of Massachusetts  
SL: NLABS, WRAIR

Various chemical reagents, known to extract spore coat protein, were used to extract spore-lytic enzyme (SLE) from intact and germinated spores of *Clostridium perfringens*. Of the reagents tested, 7.2M urea plus 10% (v/v) mercaptoethanol (UME), pH 2.85, solubilized the most SLE activity per mg spores. The quantity of SLE extracted was dependent on the starting pH of the reagent, reaching a maximum between pH 2.70 and 3.00. Germinated spores yielded more SLE than nongerminated spores upon UME extraction. SLE release during spore germination probably utilizes a trigger mechanism not satisfied by germination alone. Significant amounts of SLE were released

during germination when spores were suspended in potassium chloride or a complex germinant containing brain heart infusion, yeast extract, and chloramphenicol, but not during germination with sodium nitrite, which non-enzymatically lysed the cortical peptidoglycan. Greater solubilization of SLE activity was obtained by UME extraction of nitrite-germinated spores than of either complex germinator potassium chloride-germinated spores.

#### Reports:

1. Sensitivity of Chemically Treated Spores of *Clostridium Perfringens* Type A to an Initiation Protein, by Thomas J. Franceschini et al, *Microbios* 25, 85 (1979). AD A097 150
2. Partial Purification and Characterization of an Initiation Protein from *Clostridium Perfringens*, by Ronald G. Labbe et al, *Biochimica et Biophysica Acta*.

#### 16765 NONDESTRUCTIVE BIOPHYSICAL PROBES OF THE BASIS AND MECHANISM OF RESISTANCE IN MICROBIAL SPORES

Philipp Gerhardt  
Michigan State University  
SL: NLABS

Areas of research being pursued include: (1) The heat resistance of bacterial spores due to their partial dehydration by reverse osmosis; (2) Preparation and characterization of various salt forms of *Bacillus megaterium* spores; (3) Heat resistance of bacterial spore types correlated with wet density, water content, and inner-outer protoplast volume ratio; (4) Photometric immersion refractometry of bacterial spores; (5) Isopycnic density gradient determination of wet and dry densities of representative bacterial spore types; (6) Effect of water activity on heat resistance of vegetative cells of *Bacillus megaterium*; and (7) Absorption of ultrasound by spores and vegetative cells.

#### 17029 MECHANISMS FOR REGULATION OF THE ACTIVITY OF KEY ENZYMES IN DEVELOPING DORMANT AND GERMINATED BACTERIAL SPORES: THE ENZYMIC BASIS OF DORMANCY

Peter Setlow  
University of Connecticut Health Center  
SC: NLABS

Work has been initiated to determine when in sporulation the low internal pH of the mature spore



(6.3-6.5) is reached. If this is well before actual onset of metabolic dormancy, it could implicate a low internal pH in the latter process. Preliminary experiments have indicated that the pH of the total sporulating cell falls during sporulation. Work is in progress on the novel disulfide reductase found in *B. megaterium*. It has been shown: (1) that the enzyme is at a slightly higher level in the mother cell compared to the forespore; (2) that the enzyme is present at normal levels in an asporogenous mutant blocked early in spore germination; and (3) that the analogous enzyme is present in *B. cereus* at a 10 times higher level of activity than ever attained in *B. megaterium* but in *B. cereus* the enzyme level does not vary with the stage of growth. The latter three findings have together suggested that a major role of this novel disulfide reductase may be in the growing or sporulation cell - not only the spore, and consequently that the true substrate of this enzyme should be at a high level in sporulating cells. Therefore, a procedure has been devised for extracting and isolating only those molecules from cells which are: (a) low molecular weight and (b) contain an SH group. Studies on the purified phosphoglycerate mutase have shown that this enzyme exists in two reversibly interconvertible forms - one active and one inactive. Conversion into the inactive form is favored by low  $Mn^{++}$ , and is reversed by readdition of  $Mn^{++}$ . Studies on  $Mn^{++}$  levels - in particular attempts to isolate novel  $Mn^{++}$  chelators from spores have resumed.

## 17061 MECHANISMS GOVERNING THE TOXIGENICITY OF FOOD BACTERIA (*CLOSTRIDIUM BOTULINUM*)

Melvin W. Eklund  
Northwest and Alaska Fisheries Center  
SL: NLABS

The research objective is to identify the toxigenic determinants in *Clostridium botulinum*. The hypothesis that plasmids are responsible for toxin production in types A and B (where phage apparently do not cause toxigenicity) will be tested. Methodology will be developed to transfer plasmids from toxic cultures to nontoxigenic derivatives. Plasmids from different cell types will be characterized for degree of similarity. *Clostridium sporogenes* and proteolytic strains of *Clostridium botulinum* types A, B, and F are biochemically and physiologically closely related.

The main characteristic that differentiates the two species is the production of neurotoxin by *C. botulinum* strains. In previous studies *C. sporogenes* strains were shown to be resistant to the phages of *C. botulinum*. Because of the possibility that *C. sporogenes* might carry phages closely related to *C. botulinum* and therefore be immune to infection, several strains of *C. sporogenes* were cured of their prophages. Of the four strains of *C. sporogenes* tested, three have been cured of at least one of their prophages. Lysates of 79 strains of *C. botulinum* were tested on these cured *C. sporogenes* derivatives. Some culture supernatants of *C. botulinum* type B produced lytic or inhibitory reactions on *C. sporogenes* laves but none of these reactions were caused by bacteriophages. Filtrates from the *C. botulinum* strains were also cultured with the *C. sporogenes* derivatives, but toxicity was not induced. When *Staphylococcus aureus* is infected with specific bacteriophage, they cease to produce certain toxins. If these cultures are cured of their phages, they again produce these toxins. It is possible that a similar phenomenon occurs with *C. botulinum* and *C. sporogenes*. The *C. botulinum* strains could be infected with certain phages and become nontoxigenic and therefore be identified as *C. sporogenes*. When the *C. sporogenes* strains were cured on their phages, they continued to be nontoxigenic. These specific phages therefore do not inhibit toxin production by these three *C. sporogenes* strains. These derivatives of *C. sporogenes* were sensitive to phages of other *C. sporogenes* cultures, indicating the close relationship of these different strains. The effect of these phages on the toxigenicity of the derivatives is being tested.

## 17391 LIPID AUTOXIDATION: DEVELOPMENT AND APPLICATION OF SPIN TRAPPING TECHNIQUES TO MECHANISTIC STUDIES

Elmer J. Rauckman  
Gerald M. Rosen  
Duke University Medical Center  
SL: NLABS, CSI

The research objective is to investigate lipid autoxidation by the techniques of spin trapping free radical intermediates. Trapping techniques will be improved to provide improved qualitative and quantitative procedures for lipid alkoxyl and lipid

hydroperoxyl radicals and applied to selected autoxidation mechanisms. The studies will be extended to biological systems where lipid peroxidation may be enzyme initiated.

**Reports:**

1. Spin Trapping of Free radicals During Hepatic Microsomal Lipid Peroxidation, by Gerald M. Rosen et al, *J Biol Chem*.

**17904 PROTEIN-LIPID INTERACTIONS AND MECHANISMS OF ANTIOXIDANT ACTIVITY OF PROTEINS**

Marcus Karel  
Massachusetts Institute of Technology  
SL: NLABS

The objective of the research is to elucidate the interactions between proteins and peroxidizing lipids in foods, in order to increase the shelf life of the food. Efforts will be made to (1) determine the reactions producing protein-centered free radicals upon exposure to peroxidizing lipids, the amino acids most susceptible and the effects of micro-environment and various antioxidants, and (2) design a model system and establish conditions which minimize lipid oxidation and protein-lipid interaction.

**17910 NUTRITIONAL FACTORS AFFECTING THE INTERSTITIAL PNEUMONITIS CAUSED BY SATURATED FAT**

Samuel B. Tove  
North Carolina State University  
SC: NLABS

The research objectives are to investigate the toxicity of dietary saturated fatty acids and their monoglycerates. Determine effect of cholesterol and its oxidation products on the demonstrated toxicity of glycerol monopalmitate. Reinvestigate effects of low levels of oleate as a promotor of saturated fat toxicity. Study effects of selected amino acids and proteins on saturated fat toxicity. Standard nutritional biochemical procedures for study of dietary elements will be used. Screening studies will be done on mice, confirmatory work

**C. Environmental Quality, Protection, Rehabilitation**

will use rats and rabbits. Animals showing adverse signs will be carefully examined by consulting pathologists for morphological changes as well as basic biochemical alterations.

**18224 THE HEAT-STABLE ENTEROTOXIN(S) OF YERSINIA ENTEROCOLITIA IN FOODS**

Stephen L. Taylor  
University of Wisconsin at Madison  
SL: NLABS

The objective of the research is to evaluate the role of the heat-stable enterotoxin of *Yersinia enterocolitica* in food related illness. Efforts will be made to (1) purify the enterotoxin and determine the oral toxicity to primates, and (2) define temperature range and nutrient requirements for toxin production.

**B. PROTECTION OF MATERIAL FROM BIODEGRADATION**

**C. ENVIRONMENTAL QUALITY, PROTECTION, REHABILITATION**

**15525 THE EFFECT OF A VARIETY OF STRESSES ON THE PERSISTENCE, DETECTION & ENUMERATION OF BACTERIA OF PUBLIC HEALTH SIGNIFICANCE WITH PARTICULAR REFERENCE TO ARCTIC AND FROZEN HABITATS**

Peter H. Calcott  
Wright State University  
SC: CRREL, NLABS, AMBRDI

Several lines of approach in this study are being used. Freeze-thaw induced DNA damage, as measured by sedimentation properties of the DNA, was investigated further. The study on the plasmid and chromosome mediated resistance of *Pseudomonas aeruginosa* to freezing and thawing was continued. A *polA* strain was sensitive to ultra-violet light, x-rays and slow and rapid freezing and thawing. When the plasmid (pPL1) was introduced into the strain by generalized transduction, the resistance to each stress was increased. A mutant plasmid (pPL2), which has lost

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the UV repair gene has been transduced into the *recA* and *polA* mutants. A family of DNA repair deficient strains of *E. coli* were assessed for sensitivity to UV, x-ray and slow and rapid freeze-thaw. The reported sensitivities of the strains towards UV and x-ray were confirmed. *uvrA*, *polA*, *recA* and the double mutant *uvrA polA* strains were shown to be more susceptible to both slow and rapid freeze-thaw than either the wild-type or *lexA* mutant. The *recC* and *recB* mutants were more sensitive to slow but not rapid freeze-thaw than the wild-type. This indicates that the two major DNA repair pathways, excision repair and *rec*-dependent recombinational repair are important in determining the cryosensitivity of *E. coli*. Thus DNA could be a prime target for the stress of freeze-thaw. The study on the effects of cytoskeletal active drugs on *E. coli* was continued. Further electron-microscopy was performed to determine in more detail the events occurring in the cells.

## Reports:

- No. 1-7 in previous editions.
8. Mutagenicity of Freezing and Thawing, by Peter H. Calcott et al. *FEMS Microbiol Let* 10, 151 (1981). AD A097 838
  9. Protection of *Escherichia Coli* from Slow Freezing and Thawing by a New Class of Cryoprotectants: Ionophores, by Peter H. Calcott et al. *Cryolet* 2, 48 (1981). AD A098 723
  10. The Role of DNA Repair Genes and an R-Plasmid in Conferring Cryoresistance to *Pseudomonas Aeruginosa*, by Donna L. Williams et al. *J General Microbiol.*
  11. Transient Loss of Plasmid Mediated Mercuric Ion Resistance After Freezing and Thawing of *Pseudomonas Aeruginosa*, by Peter H. Calcott, *Ohio J of Sci.*

## 15992 PLANT POLLUTION INDICATOR SYSTEM - BASIS OF SENSITIVITY/ TOLERANCE TO NO<sub>2</sub>, SO<sub>2</sub>, O<sub>3</sub>

John M. Skelly  
Virginia Polytechnic Institute and State University  
SC: CSI

Eight clones of eastern white pine (*Pinus strobus* L.) with predetermined sensitivity to air pollution were fumigated in summer of 1979 and 1980 with ozone (O<sub>3</sub>), sulfur dioxide (SO<sub>2</sub>), and nitrogen dioxide (NO<sub>2</sub>), singly, and in combinations. In 1979, 8 clones were fumigated with these 3 major pollutants at 5 parts per hundred million (pphm) and 10 pphm for 4h/day for 35 consecutive days. During this fumigation period, needle length, visible symptom, and dry wt/fasicle were evaluated weekly. In 1980, 7 out of the 8 clones were fumigated at 10 pphm singly and in all

combinations for 4h/day for 56 days. The same response parameters as in 1979 were evaluated at 35th and 56th day of the fumigation. Needle length was not significantly different among clones and treatments, whereas, there was a correlation between needle dry wt and air pollution injury. Three clones of eastern white pine with predetermined sensitivity to air pollution (sensitive, intermediate and tolerant respectively) were fumigated with 10 pphm ozone (O<sub>3</sub>) and 10 pphm sulfur dioxide (SO<sub>2</sub>) for 4 hr/day for 6 consecutive days. The carbon dioxide exchange rate (CO<sub>2</sub>ER) of a single branchlet from each grafted scion was measured with an infrared gas analyzer hourly during, and 1 hr prior to and after the daily fumigation. O<sub>3</sub> and SO<sub>2</sub> decreased apparent CO<sub>2</sub>ER in all 3 clones during the fumigation periods. CO<sub>2</sub>ER recovered after the termination of each day's fumigation. The magnitudes of CO<sub>2</sub>ER decrease and the rates of reduction or subsequent recovery varied among clones and pollutant treatments. There was a trend towards continuous decrease in relative CO<sub>2</sub>ER over the 6 day period in the sensitive clone while the tolerant clone exhibited a continuous increase. The measurement of CO<sub>2</sub>ER appears to be a reliable indicator of clonal sensitivity to air pollution. In 1980, transpiration and respiration as well as CO<sub>2</sub>ER rate of 4 EWP clones at 0, 10, 20, and 30 pphm O<sub>3</sub>, SO<sub>2</sub>, and NO<sub>2</sub> used singly were measured weekly during the 4h/day, 9 consecutive weeks of fumigation. Collected data is still being analyzed.

## Reports:

No. 1-5 in previous editions.

## 16113 SURVIVAL OF MICROORGANISMS IN NATURE

L. E. Casida, Jr.  
Pennsylvania State University  
SL: NLABS SC: NLABS

The gram negative bacterial predators used previously have been designed "NYA," "7A," and "A" (which is *Flexiligradius*). Extensive physiological, growth, and phage-typing studies were done for these organisms. They are definitely different from each other, with strain "NYA" being quite different. Based on relative amounts of slime produced by its colonies, strain "A," with low frequency, produces two variants. One of these, variant "SA," is genetically stable, once produced.

Strain "A" and its two variants apparently do not differ from each other in any aspect other than slime production. Strains "A," "NYA" and "7A" all track streaks of host cells, but do not do so if initially applied adjacent to but not touching the host cell streak. Thus, there does not seem to be a diffusible chemotaxis activator. On an agar medium containing high carbon but very low nitrogen levels, strains "A," "NYA" and "7A" produce some kind of diffusible substance that kills *M. luteus* host cells. However, production of this material in broth has been unsuccessful to date. The survival of *Arthrobacter oxydans* in natural soil (not in presterilized soil as is usually done) was studied. This was accomplished by using a nicotine-salts selective plating medium. Cystites and coccoid cells in this soil survived equally well, and a slow drying of the soil had little effect. However, multiplication of the cells in soil, as opposed to just surviving, occurred only with glucose or nicotine addition to the soil; multiplication did not occur with a range of other carbon and nitrogen nutritive additions. The production in natural soil of indigenous (not added) bacteriophage for various *Arthrobacter* species was studied. The *Arthrobacter* host cells were not added to the soil but were naturally present. The procedures developed for this study, and the results obtained, predicted that a technique using phage could be developed for following the survival and activities of various other bacteria, or their bacterial predators, in soil. Studies on *Azotobacter* cyst production and the survival of myxogacteria and *Ps. aeruginosa* in soil are continuing.

#### Reports:

- No. 1-3 in previous edition.
4. Myceloid Growth of *Arthrobacter globiformis* and Some Other *Arthrobacter* Species, by James J. Germida et al, *J Bacteriology* 144, 1152 (1980). AD A095 904
  5. Survival of *Arthrobacter oxydans* in Natural Soil, by James J. Germida et al, *Can J. Microbiol.*
  6. *Arthrobacter* Bacteriophage from Soil, by James J. Germida et al, *J Appl Environ Microbiol.*

#### 16592 REVEGETATION OF ALASKAN DISTURBED SITES BY NATIVE TUNDRA SPECIES

F. Stuart Chapin, III  
Gaius R. Shaver  
University of Alaska  
SL: CRREL

The survey of vehicle trails in Alaskan tundra has been completed and several conclusions have been

drawn concerning differences in nutrient cycling between trails and undisturbed tundra. Vehicle trails are somewhat warmer (2C) than control soils and in some cases have higher phosphate availability, but these differences in temperature and nutrients are insufficient to explain the 10-15-fold higher annual nutrient accumulation by plants in the trails. It has been hypothesized that differences in rate of water movement are in part responsible for nutritional differences between trails and controls by moving nutrients to the root surface and overcoming normal diffusion limitation of uptake. A simulation model suggests that a water movement rate of only 1-2 mm per day can be extremely important in overcoming diffusion limitation and increasing plant growth. In certain trails iron becomes reduced from ferric to ferrous state, releasing phosphorus into the soil solution. Soil moisture also plays a key role in nutrient mineralization. In 1979 there was higher soil phosphatase activity in the wet than the mesic site. In 1980 mesic soils were wetter, and both soil moisture content and soil phosphatase activity were similar between sites. There were no differences in  $K_m$  of soil phosphatase between track and control sites, suggesting that the differences in activity between these sites results from differences in quantity or activity of enzymes rather than any major qualitative differences. Results of 4 years' work on artificially disturbed tundra sites are now being summarized. *Eriophorum vaginatum*, the dominant species of tussock tundra maintains itself in the natural community by establishing in small naturally occurring disturbances such as frost boils. It is thus well adapted to disturbance and also successfully establishes in man-made disturbances.

#### 16601 MICROBIAL GROWTH AT ULTRASLOW RATES: REGULATION AND GENETIC STABILITY

William Chesbro  
University of New Hampshire  
SC: WRAIR

Growth in the recycling fermentor always follows a 3-stage pattern: an initial stage of batch-type growth, a second phase of slower, linear growth whose length is independent of growth rate and is characteristic of the strain employed, and a third phase of still slower growth and of indefinite length.



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D. Defense Against Chemical and Biological Weapons

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In the second and third phases the specific growth rate, and the doubling time continuously fall and increase respectively until, in phase 3, doubling times of hundreds of hours are reached. In phases 2 and 3 cellular levels of the regulatory nucleotide guanosine tetraphosphate (ppGpp) increase, reaching a maximum, steady concentration at the start of phase 3 and thereafter. This nucleotide is the major factor in the stringent regulatory response, affecting biosyntheses at the translation, transcription, and enzymatic levels. The hallmark of the stringent response is the interdiction of rRNA synthesis and lowered cellular proportions of RNA, which is also the characteristic feature of phase 3 in the recycling fermentor. The synthesis and degradation of ppGpp is under the control of the products of 4 genetic loci: the *rel A* gene product synthesizes quanosine pentaphosphate, a precursor of ppGpp, at the ribosome in the presence of mRNA and uncharged transfer RNA, thus tying its synthesis to amino acid deficiency; the *rel X* and *rel S* products are still obscure in the details of their action, but they bring about the synthesis of ppGpp directly when the cell experiences an energy shortfall; and the *spot* gene stipulates a pyrophosphatase degrading ppGpp to guanosine diphosphate and pyrophosphate. Thus, the cellular level of ppGpp is the summation of the activities of these gene products.

#### Reports:

No. 1 in previous edition.

## 17512 REGULATION AND CHARACTERIZATION OF CELLULASE BIOSYNTHESIS IN THERMOMONOSPORA CURVATA

Fred J. Stutzenberger  
Clemson University  
SL: NLABS

The research objective is to increase production of cellulase by *thermomonospora curvata* under conditions suitable for eventual military or industrial use. Efforts will be made to: (1) Purify the exoglucanase and characterize the action on cellulase polymers. (2) Enhance cellulase production by mutagenesis and develop a selective medium to isolate derepressed mutants. (3) Determine relationship of cyclic AMP to regulation of cellulase biosynthesis.

## 17823 RAINFALL CHEMISTRY & POTENTIAL BENEFICIAL/ DETRIMENTAL IMPACT ON INDIGENOUS VEGETATION

John M. Skelly  
Virginia Polytechnic Institute and State University  
SC: CRREL, AMBRDL, USATHAMA

The research objective is to analyze rainfall chemistry at field sites related to emissions from the Radford Army Ammunition Plant (RAAP), Virginia and determine downwind characteristics in relation to other rainfall in the region. The effect of analyzed precipitation on selected indicator tree species in field environments will be determined. The research will include: (1) Collecting simultaneous samples of rainfall and air from sites on and off RAAP property related to emissions from explosive production lines and the power plant, as well as upwind standard samples, for concentrations of NO<sub>x</sub> and SO<sub>2</sub> and hydration products. (2) Assaying leaf or needle samples from indicator trees for acute and chronic effects on physiology of leaf tissue. Gross and microscopic effects will be examined.

#### Reports:

1. Variation in the Physiological Processes of Eastern White Pine (*Pinus Strobus L.*) to Ozone, Sulfur Dioxide, and Nitrogen Dioxide, by Yaw-Shing Yang, May 81, 162p, figures, PhD Thesis.

## D. DEFENSE AGAINST CHEMICAL AND BIOLOGICAL WEAPONS

## 15464 A PHYSIOLOGICAL AND BIOCHEMICAL BASIS FOR THE ACTION OF SOMAN AND RELATED AGENTS AT THE ACETYLCHOLINE RECEPTOR

Francis C. G. Hoskin  
Illinois Institute of Technology  
SL: CSL

A DFP-hydrolyzing enzyme has been attached to Sepharose 6B resin with cyanogen bromide. In a typical preparation, 82% of a highly purified soluble DFPase is converted to the immobilized enzyme, and 18% remains in solution. The high

stability of the DFPase is the main factor in this quantitative accounting. After extensive washing the Sepharose-DFPase showed approximately the same kinetic properties as the soluble enzyme. The immobilized enzyme is also extremely stable, even when dried for several months, although it should be noted that the dried enzyme was stored cold. A 0.7 x 50 mm column of the immobilized enzyme has been prepared and a buffered solution of DFP passed through it at a rate of 0.1 ml per min with a 50 mm aqueous pressure head. At  $10^{-4}$  or  $10^{-3}$  M DFP applied, both fluoride electrode measurements and the inhibition of AChE by the column effluent showed that more than 99% of the DFP had been hydrolyzed. When the same experiments have been performed with Soman, fluoride electrode measurements show greater than 90% yield of fluoride, again suggesting nearly complete hydrolysis. On the other hand, AChE inhibition measurements on the column effluent show consistently that about half of the AChE-inhibiting power remains. Furthermore, when this column effluent is recycled through the immobilized-enzyme column, more than 80% of the AChE-inhibiting power emerges undiminished. These results pose the possibility of a serious hazard, and a dilemma, namely, that when nearly all of the Soman is hydrolyzed (P-F bond splitting) about half of the AChE-inhibiting power remains.

#### Reports:

1. A Description and Analysis of Novel Conductance Events in *Electrophorus* Electropaque, by Donald A. Farquharson, *J Neurosci*.
2. Penetration of VX into Nerve Cells, and Effects on Electrical Function, by Donald A. Farquharson et al, *Bull Environ Contamntn and toxiclgy*.

#### 15761 THE ACETYLCHOLINE RECEPTOR AND ITS IONIC CHANNEL AS TARGETS FOR DRUGS AND TOXINS

Mohyee E. Eldefrawi  
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University of Maryland School of Medicine  
SC: CSL

Chemostat populations of *Escherichia coli* were grown under lactose, glycerol and ammonium-limited conditions at growth rates between 0.1 and

0.5h<sup>-1</sup>. When in steady state, the cells were examined for cyclic AMP, cyclic GMP levels and four enzyme levels (glucose-6-phosphate dehydrogenase, isocitrate dehydrogenase, NADH oxidase and phosphodiesterase). All four enzyme activities correlated with both cyclic nucleotide levels determined over all the growth conditions. However, each enzyme activity appeared to be related to the level of nucleotides in a distinct fashion. Exogenous cyclic AMP, cyclic GMP and phencyclidine modulated the levels of phosphodiesterase and isocitrate dehydrogenase in a manner predictable from the correlations. A "Permutation Hypothesis" was proposed to account for the ability of bacteria to regulate metabolic functions under diverse growth conditions. A temperature-sensitive mutant of *E. coli* was isolated which was able to grow only poorly at 37C; its growth at 25C was normal. The mutant, unlike the parent, was supersensitive to exogenous cyclic GMP. Cyclic AMP was ineffective with both strains. Phencyclidine, which lowered cyclic GMP and cyclic AMP levels in chemostat populations, partially alleviated the effect of this mutation at 37C. The mutant was unable to lower its cyclic GMP levels at 37C, unlike the wild type. It has been proposed that the mutation caused a decrease in activity of the phosphodiesterase level or release mechanism, increased the cellular guanylate cyclase activity, deregulated the cells cyclic GMP metabolism activity, deregulated the cells cyclic GMP metabolism or associated activities.

#### 15947 HYPERTENSIVE EFFECT OF BRAIN ACETYLCHOLINESTERASE INHIBITION

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SC: DPG

Investigations have continued on the role of brain acetylcholine in the modulation of cardiovascular reflexes and in the development and maintenance of hypertension. Activation of central cholinergic mechanisms is achieved by means of acetylcholinesterase inhibitors while centrally injected hemicholinium-3 is employed to inhibit the synthesis of brain acetylcholine. The first phase of a study on the involvement of brain acetylcholine

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D. Defense Against Chemical and Biological Weapons

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in the modulation of reflex bradycardia and tachycardia has been completed. The results confirm preliminary findings that brain acetylcholine enhances reflex bradycardia and inhibits reflex tachycardia evoked, respectively, by increases and decreases in blood pressure. The modulatory effects are produced in unanesthetized rats by intracerebroventricular injections of the acetylcholinesterase inhibitor, physostigmine. The effects of physostigmine can be prevented by prior administration of atropine or by depletion of brain acetylcholine stores by hemicholinium. Considerable progress also has been made in understanding the mechanisms by which inhibition of brain acetylcholinesterase potentiates the pressor response to occlusion of the carotid arteries. Results have also been obtained in experiments of the role of brain acetylcholine in hypertension.

## Reports.

No. 1-5 in previous editions.

## 16146 TRANSITION STATE ANALOG INHIBITORS FOR ESTERASES

Alan Daffron  
Bowling Green State University  
SL: CSL SC: WRAIR

**Reversible Acetylcholinesterase Inhibitors:** Work on boronic and borinic acids as transition state analogs has been completed.  $C_6H_5B(OH)CH_3$  has been shown to block the irreversible inactivation of the enzyme by  $CH_3SO_2F$ . This protection confirms our hypothesis that the boron compounds form a complex with the Serine-OH of the enzyme. Inhibition by compounds such as  $o-CH_3OC_6H_4CH_2B(OH)_2$  was found to be slightly time dependent, providing further evidence for coordination with serine. **Irreversible Acetylcholinesterase Inhibitors:** Studies on irreversible inhibition have concentrated on better synthetic methods and on improving the reproductibility of the rate measurements to allow studies of pH dependence. Experiments to see whether reversible inhibitors will protect the enzyme from irreversible inactivation by  $CF_3SO_2Cl$  have produced interesting but unexpected results. The extent and kinetic form of the protection observed appears to vary depending on

the reversible inhibitor used. Work to resolve these discrepancies is continuing.

## Reports:

1. Substituent Effects on Inhibition of Acetylcholinesterase by Ketone Transition State Analogs, by Alan Dafforn et al, *J Biol Chem*.
2. Inhibition of Collagenase by Anti-Arthritic Gold (I) Compounds, by Julie M. Finke et al, *Biochimica and Biophysica Acta*.

## 16583 MOLECULAR CHARACTERISTICS OF MEMBRANE GLUTAMATE RECEPTOR-IONOPHORE INTERACTION

Elias K. Michaelis  
University of Kansas  
SL: CSL

Previous studies indicated that binding of glutamic acid to its receptor sites may induce an electrogenic  $Na^+$  flux which then results in membrane depolarization. It has been demonstrated that L-glutamate indeed produces  $Na^+$  fluxes across synaptic membrane vesicles. The electrogenicity of the L-glutamate-stimulated  $Na^+$  fluxes has been explored. The rate of  $^{35}SCN^-$  uptake was increased by 3.3 mM  $NaN_3$  which is a good inhibitor of the L-glutamate binding process, but the maximum uptake remained about the same. The effect of  $NaN_3$  is very similar to that of the L-glutamate effect. Stimulation of  $^{35}SCN^-$  uptake by L-glutamic acid was not observed in the presence of  $NaN_3$ . A good inhibitor of the L-glutamate carrier system, p-hydroxymercury benzoic acid, did not change the pattern of passive  $^{35}SCN^-$  uptake. However, it increased further  $^{35}SCN^-$  uptake activity in the presence of L-glutamate. This seems to exclude the possibility that the known L-glutamate carrier process contributes to some extent in the membrane potential change induced by L-glutamate.

## Reports:

- No. 1 in previous edition.
2. Effects of L-Glutamic Acid on Synaptosomal and Synaptic Membrane  $Na^+$  Fluxes and  $(Na^+-K^+)$ -ATPase, by Hsuan H. Chang et al, *J Biol Chem* 255, 2411 (1980). AD A091 368
  3. L-Glutamate Effects on Electrical Potentials of Synaptic Plasma Membrane Vesicles, by Hsuan H. Chang et al, *J Biol Chem*.

**18034 SYNAPTIC NEUROCHEMISTRY OF  
SENSORY PATHWAYS IN THE  
INSECT CENTRAL NERVOUS  
SYSTEM**

John Hildebrand  
Columbia University  
SL: BML

The objective of the research is to study cellular distribution, metabolism, and membrane receptor systems for key neurotransmitter molecules and neuromodulators in *Manduca sexta* moths as a model system for central nervous system (CNS) structures and functions. Focus will be on visual and chemosensory systems. Transmitter chemicals sought include acetylcholine, gamma-aminobutyric acid, octopamine, serotonin, dopamine, glutamate and aspartate. Previous knowledge of cellular organization, physiology and development of neuropathways will serve as basis for survey of neurochemistry of synaptic systems. Radiochemical techniques, HPLC, autoradiography, histofluorescence, and ligand-binding techniques will be used.

**18126 CHEMORECEPTOR FUNCTION AND  
REGULATION IN CULTURED  
VERTEBRATE NEURONS**

Jeremy B. Tuttle  
University of Connecticut  
SL: BML

The research objective is to study regulation of chemosensitivity and its relation to neurotransmitter chemicals by synaptic systems in vertebrate neurons in tissue culture. Information developed in homogeneous cultures will be tested in cultures of interactive mixed cell types. Tissue cultures of homogeneous and mixed interactive nerve cells will be used as models for study of function, regulation and pharmacology of neurotransmitters by means of standard neurochemistry and neurophysiology techniques. Potential study materials include: 5-hydroxytryptamine, dopamine, norepinephrine, gamma-aminobutyric acid, glycine, glutamate, neuroactive peptides.

**E. Sensory Factors in Performance Enhancement**

**18241 THE PHYSIOLOGY,  
BIOCHEMISTRY AND GENETICS  
OF SURVIVAL OF BACTERIA  
SUBJECTED TO ENVIRONMENTAL  
STRESS**

Petter H. Calcott  
Wright State University  
SC: NLABS, CRREL

The objective of the research is to improve methodology for the detection and enumeration of potential pathogens and other microorganisms in the environment. Attempts will be made to: (1) Determine the effects of altering wall and membrane components on stress related injuries, both phenotypic and genetic alterations will be used. (2) Investigate the requirements for repair of injuries by the cells, and the kinetics of repair in natural environments. (3) Determine all major types of cell damage which could occur and document which of these are capable of repair.

**E. SENSORY FACTORS IN PERFORMANCE  
ENHANCEMENT**

**16729 EFFECTS OF IMPULSE REPETITION  
OF DETECTABILITY OF  
HELICOPTER-LIKE ACOUSTIC  
SIGNALS**

Sanford Fidell  
Bolt Beranek and Newman, Inc.  
SL: AMES, ARL

Measurements of the detectability of low frequency signals masked by noise were made under several conditions. Signal duration, repetition rate, and spectral shape of signal and noise were varied in four different studies. All four studies shared common means of solving the technical problems associated with the control and specification of low frequency signals. The data from the studies support the inference that effecting masking bandwidths (critical bands) increase for signal frequencies below 125 Hz, and that for these low frequencies, the shape of the hypothetical auditory filter is essentially symmetric on a linear frequency scale.

**Reports:**

1. Effects of Impulse Repetition on Detectability of Helicopter-Like Acoustic Signals, by Sanford Fidell, Dec 80, 7p, Annual TR: 1 Feb 80 - 30 Nov 80.



2. Detectability and Annoyance of Repetitive Impulsive Sounds, by Sanford Fidell et al, *Proc 37th Annu Forum and Technology Display*.

# 16867 BIOCHEMICAL STUDIES ON THE INITIATION OF ODOR SENSING

Robert B. Koch  
Mississippi State University  
SC: MERADCOM, CSL

The objective of the research is to determine the nature of interaction between tissue membrane functions and chemical odorants which cause differential perturbations of membrane bound  $\text{Na}^+/\text{K}^+$  ATPase activity. At present the program consists of the following approaches: (1) Investigation of left-right symmetry of response of  $\text{Na}^+/\text{K}^+$  ATPase activity from individual turbinal preparations from rat olfactory tissue; (2) Effect of an antibody to an anisole binding protein from dog olfactory tissue on odorant perturbation of  $\text{Na}^+/\text{K}^+$  ATPase activity from cow olfactory tissue preparations; (3) Studies of cow olfactory tissue preparations, including NEP-mitochondrial preparations from individual turbinals and from combinations of turbinals from the whole olfactory epithelial tissue; and (4) Initiation of studies on enzyme purification from cow brain.

## Reports:

1. Odorant Responses of  $\text{Na}^+/\text{K}^+$  ATPase Activity by Preparations from Paired Turbinals of Rat Olfactory Tissue, by Herman Rossi et al, *Chem Senses*.
2. Effect of Antibody to Anisole Binding Protein on Odorant Perturbation of  $\text{Na}^+/\text{K}^+$  ATPase Activity, by R. B. Koch et al, *Chem Senses*.
3. *In Vitro* Response of ATPase Activities in Tissue Subcellular Particle Preparation to a Series of Mono-Unsaturated C<sub>18</sub> Fatty Acids, by Robert B. Koch *Biochem Pharm.*

# 17085 BRAIN MECHANISMS UNDERLYING INDIVIDUAL DIFFERENCES IN REACTION TO STRESS: AN ANIMAL MODEL

Jerome Siegel  
University of Delaware  
SL: HEI SC: WRAIR

The research objective is to investigate the electrophysiology of evoked potentials from

control areas of the brain of animals under behavioral stress; investigate neuronal mechanisms at the cellular level that cause the observed augmenting - reducing behavioral traits; explore the genetic and early-experience determinants of such behavior. Studies will use cats for the evoked potential and implanted electrode studies on stress reaction. Stimuli used in evoked potential studies will be light flashes. Single cell recordings will be taken from light flash stimulus of varying intensity to map response relation to evoked potential mechanisms. Genetic and early-experience studies will be performed on selected strains of rats. The first experiment conducted was designed to find out if the rat is a suitable subject for evoked potential augmenting - reducing experiments. It was found that rats, like cats, can be divided into two categories in terms of their reactions to certain environmental conditions such as novelty stress. Some rats are highly reactive; others are minimally so. Most relevant is the finding that these two populations are also distinguishable in terms of their visual cortical evoked potentials. It appears that the rat, as well as the cat, can serve as an animal model for investigating the neurophysiological differences in reaction to stress. The use of the rat will readily permit exploration of the genetic and early experience determinants of these individual differences.

## Reports:

1. Electrophysiological and Behavioral Correlates of Arousal, by Rhawn Joseph et al, *Physio Psych.*

## F. BIOLOGICAL SCIENCES PROGRAM

# 15319 IMPORTANCE OF EXTRACELLULAR PROCESSES IN THE OXYGEN ENHANCEMENT OF RADIATION LETHALITY

Irwin Fridovich  
Duke University  
SL: NLABS SC: CSL, NLABS

The unusual situation discovered in *Lactobacillus plantarum* where mM levels of Mn(II) takes the

place of M levels of SOD in protecting the cells against  $O_2^-$ , has been further explored. A wide range of *Lactobacilli* have been surveyed. Those which ordinarily live in Mn-rich environments, such as fermenting plants, contain high levels of Mn(II) and no SOD. In contrast those which live in milk or other Mn-poor media, have SOD and do not have high Mn(II). No case was found of a variety which had both defenses against  $O_2^-$ . When a naphthoquinone (plumbagin) was used to cause increased intracellular production of  $O_2^-$  it could be shown that high Mn(II) or SOD were capable of providing protection. The search for a stable SOD mimic continues. *Photobacter leiognathi*, which lives as a symbiont in the ponyfish, is very peculiar in that it contains CuZnSOD which is usually found only in eukaryotes. A very close relationship has been shown between the CuZnSOD of *P. leiognathi* and that of the ponyfish. Indeed a computer-assisted comparison of the amino acid compositions of sixty different superoxide dismutases showed that the *P. leiognathi* SOD was much more closely related to teleost fish SOD than to SOD's from any other group of living things. A gene transfer from the fish to its bacterial symbiont is strongly supported by these data. The popular conception of obligate anaerobes is that they do not encounter  $O_2$  and need no defenses against it. Yet, such organisms must survive aerobic exposure enroute from one anaerobic niche to the next. The methanogens have been chosen for study because of their almost legendary oxygen-intolerance and because they are reputedly a very primitive group of organisms.

#### Reports:

No. 1-23 in previous editions.

24. Defenses Against Oxygen Toxicity in *Lactobacillus Planarum*, by Frederick S. Archibald et al, *J Bacteriology*.
25. Evidence for a Natural Gene Transfer from the Ponyfish to its Bioluminescent Bacterial Symbiont, *Photobacter Leiognathi*: The Close Relationship between Bacteriocuprein and the Copper-Zinc Superoxide Dismutase of Teleost Fishes, by Joseph P. Martin, Jr. et al, *J Biol Chem*.
26. Manganese, Superoxide Dismutase and Oxygen Tolerance in Some Lactic Acid Bacteria, by Frederick S. Archibald et al, *J Bacteriology*.
27. Isolation and Characterization of the Iron-Containing Superoxide Dismutase of *Methanobacterium Bryantii*, by Thomas W. Kirby et al, *Arch Biochem Biophys*.

28. Continuous Colorimetric Monitoring of the Fructose Biosphosphate Aldolase Reaction, by Philmore Robertson, Jr. et al, *Anal Chem* 108, 332 (1980). AD A095 493
29. Manganese and Defenses Against Oxygen Toxicity in *Lactobacillus Plantarum*, by Frederick S. Archibald et al, *J Bacteriology* 145, 442 (1981). AD A096 916

#### 15893 INVESTIGATION OF THE MECHANISMS IN FIELD-DESORBED ION FORMATION USING A PULSED FIELD DESORPTION TIME-OF-FLIGHT MASS SPECTROMETER

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SRI International  
SL: NLABS SC: CSL

The objective of the research has been the development of pulsed field desorption time-of-flight mass spectrometry as a sensitive analytical instrument for the detection and identification of biological materials. A mass spectrometer has been designed, built and tested. A facility for producing silicon whisker activated field emitters has been developed. These new emitters are easily and rapidly activated and readily ionize compounds with relative high ionization potential. This latter fact indicates that the electric field strength near the sharply pointed tips of the silicon whiskers is very high. The mass spectrometer has now reached the stage where it may be operated easily, routinely, and reliably. Efforts are being concentrated on the study of the time dependence of ion formation during the field desorption process. The two important factors appear to be the supply of sample molecules or ions to the field desorbing sites and the rate of field desorption itself from these sites. For species such as alkali metal cations, the rate of supply is much greater than the rate of desorption, with the result that the shape of the ion pulse closely follows the shape of the high voltage field desorbing pulse, regardless of the pulse width. However, for the formation of protonated molecular ions from species such as arginine, the rate at which ions or molecules are supplied to the field desorbing sites is slow compared to the rate of field desorption. Therefore, most of the ions are desorbed at the beginning of the high voltage pulse. Emphasis is

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being placed on studies of the field adsorption process.

*Reports:*

No. 1 in previous edition.

## 16272 ELECTRORETINOGRAM (ERG) IMPLICIT TIME

John B. Siegfried  
Pennsylvania College of Optometry  
SL: HEL SC: NVEOL

Efforts are continuing to maximize the recording of the electroretinogram (ERG) through the use of specially modified scleral contact lenses. The current model is adequate and is well tolerated by the volunteer student subjects. Because of the improved signal-to-noise ratios achieved by this recording device, measurements have been obtained which are close to psychophysical threshold for test flashes of various wavelengths superimposed upon steady adapting backgrounds which are either white (Xenon) or yellow (585 nm). A useful adjunct recording technique has been found for measuring very low amplitude ERG's elicited under these near-threshold conditions. This is to selectively filter the amplified ERG signals with active analogue filters having a passband of 60-200 Hz. By thus eliminating the low frequencies in the ERG recordings, the system gain can be increased, and the minute changes recorded in a form which enables more accurate measurement of implicit time (latency from flash onset to response peak). In order to compare data obtained by the present technique of superimposing a small area spectral test flash upon steady adaptation background, with data obtained by the technique of alternating barred patterns, which eliminate the effects of stray light, several additions are being made to the optical system.

## 16689 RESEARCH IN THE AREA OF VISION AS RELATED TO SEARCH AND CAMOUFLAGE

David Sheena  
G&W Applied Sciences Laboratories  
Partially Funded by HEL  
SL: HEL, WRAIR SC: NVEOL, ARI

**BRIGHT PUPIL EYE POSITION MEASUREMENT SYSTEM:** Work continued on this area, i.e. integrating this system with the existing Human Engineering Laboratory (HEL) oculometer. A mirror system was added for tracking the movement of the subject's head. Remote motorized focusing capability was installed to allow for subject distance changes. Thus far, the mechanical, optical and illumination constraints of the two systems have been kept compatible with each other so that they can operate independently or simultaneously. The two calibration procedures each suitable for one of the systems, are being tested. The two procedures are different only in the number of scene calibration points and in the particular transformation algorithm employed. Two units are intended to produce two supplementary sets of eye position measurements and work is in progress to interface data from the bright pupil measurement system into the HEL computer. Both sets of measurements will be stored for later analysis and comparison to determine the optimal types of subject for each system. **STIMULUS GENERATION:** The visual tracking experiments require a large number of different slides reflecting various conditions. Generating slides with elements that reproducibly fall at the same points on the screen has always been a difficult task. A positionable camera support along with special slide mounts have been assembled so that this job can more easily be done at HEL.

### III ENGINEERING

#### A. SOLID MECHANICS

##### 14760 RESPONSE OF A THICK-WALLED TUBE HAVING SLIGHT INITIAL CURVATURE TO TRANSIENT INTERNAL LOADINGS

W. A. Nash  
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SL: ARRADCOM, BWL SC: AMMRC

The elastodynamics of a thick-walled tube subject to transient internal pressure loading acting uniformly around the inner boundary of the tube is being considered through use of the method of characteristics. A computer code has been developed (implemented for CDC in Fortran IV language) that predicts stresses and velocities at points in a grid-work in the thick tube. The loading is a transient internal pressure applied along the internal boundary of the tube and extending uniformly from one end of the tube to the other. The loading is taken to be axisymmetric along the length. The method of characteristics has successfully been employed to solve the linear elastodynamic equations characterizing the response of the tube to this transient pressure. The sequence of events is that the pressure is first applied to the inner boundary and then various types of pressure waves propagate from that cylindrical boundary to the outer cylindrical boundary as well as the flat ends of the tube. These waves then reflect back from these surfaces into the interior of the tube. The computer program gives resultant stress fields due to the initial as well as reflected waves. It would be possible to generalize this approach to include the next step to reflections from the internal boundary outward again.

*Reports:*  
No. 1 in previous edition.

##### 14932 THE VALUE OF DYNAMIC FRACTURE TOUGHNESS AND ITS DEPENDENCE ON MATERIAL PROPERTIES AND LOADING CONDITIONS

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Stanford University  
SL: AMRDL, ARRADCOM, SC: MICOM, BRL  
AMMRC

Six manuscripts are being prepared: (1) Correction to "The Effect of Axial Force on Dynamic Fracture of a Beam or a Plate in Pure Bending", (2) Dynamic Fracture of a Beam or Plate Under Tensile Loading, (3) Symmetric Dynamic Fracture of a Beam or Plate Under Tensile Loading, (4) Effect of Bending Moment on the Dynamic Fracture of a Beam or Plate under Tensile Loading, (5) Effect of Shear and Rotary Inertia on Dynamic Fracture of a Beam or Plate Under Tensile Loading, and (6) Effect of Shear and Rotary Inertia on Dynamic Fracture of a Beam or Plate in Pure Bending. In all, it was found that the models adequately describe the fracture process until wave reflection effects come into play. A model being investigated at the present time is a long finite-length Timoshenko beam. It is expected that this model will provide the preliminary interaction effects of fracture and wave reflection needed to describe the fracture of the final ligament of the beam or plate.

*Reports:*  
No. 1-2 in previous edition.

##### 15078 STRESS FIELD EFFECTS ON DYNAMIC FRACTURE

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SL: AMMRC, BRL SC: BWL, ARRADCOM

Studies have indicated that the strain at failure can be predicted under both static and dynamic loading conditions for thin rings. In addition it was determined that the same theory could be applied to predict the minimum stress level required for



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spallation under the high strain rate conditions associated with plate impact. This theory has been extended to include the plane-strain thick walled cylinder case, and the necessary computer programming is now being undertaken. The material properties that relate to the failure process in the above analysis is the critical void volume level. This property can be determined from static ring tests, but the verification of its application to dynamic conditions can best be performed using the impacted ring configuration. The experimental facility to carry out these tests on high strength steel specimens has been designed and tested.

## 15226 ELASTIC-PLASTIC DEFORMATION IN CRACKED SOLIDS AND DUCTILE CRITERION

H. W. Liu  
Syracuse University  
SL: APG, BWL, ARRADCOM

When a cracked solid is stressed, the crack tip opens up and it propagates ahead. Crack tip opening displacement, CTOD, is directly related to  $J$ , and it has been widely used for ductile fracture analyses under monotonically increasing load. Under a cyclic fatigue load, a crack may propagate by brittle fracture mode or propagate by ductile deformation mode. Under ductile deformation mode, crack tip opening displacement has been related to crack propagation per cycle and it has been assumed to be equal to the striation spacing. The alternating slip on two sets of mutually intersecting conjugate slip bands has been proposed as the physical process of striation formation under a cyclic load. The CTOD that blunts a crack tip and the unzipping crack increment have been calculated. The calculations compare extremely well with experimentally measured stretch zone width and striation spacing. The stretch zone width is directly related to the total CTOD that blunts the crack tip.

## 15243 DYNAMIC SIMULATION OF COMPLEX STRUCTURES

Leonard Meirovitch  
Virginia Polytechnic Institute and State University  
SL: EUSTIS

Further progress has been made in placing the substructure synthesis method on a solid

mathematical foundation. Previous work has concentrated on developing the concept of admissible vectors for discrete substructures, which is the counterpart of the concept of admissible functions for continuous substructures, and on the problem of satisfaction of geometric compatibility at boundaries between any two adjacent substructures. The more recent and current effort is concerned with generating improved sets of admissible functions (vectors). Several papers are being prepared in this area.

### Reports:

No. 1-2 in previous editions.

3. A General Dynamic Synthesis for Structures with Discrete Substructures, by L. Meirovitch et al, *Proc AIAA/ASME/ASCE/AHS 21st Structures, Structural Dynamics and Mater Conf.*
4. On the Substructure Synthesis Method, by L. Meirovitch et al, *Proc Intl Conf on Recent Advances in Structural Dynamics.*

## 15400 DYNAMIC ELASTIC-PLASTIC RESPONSE OF BEAMS IN COMBINED THRUST AND BENDING

J. D. Colton  
A. L. Florence  
SRI International  
SL: BRL SC: AMMRC, MICOM

The primary objective of this work is to analyze the elastic-plastic response of a beam in combined thrust and bending. A secondary objective is to explore the implications of this solution for the response of long-rod penetrators. A theory has been developed for predicting the response of an elastic-plastic beam in combined thrust and bending. The theory has six governing equations, which were solved using the method of characteristics. The solution is being implemented in a computer program. A major portion of the effort was devoted to developing the program logic and writing the computer program. An important aspect of this project is to determine the simplest approach that will give a solution with sufficient accuracy to be practical. Therefore, the program is being developed at three levels of complexity: (1) for elastic response, (2) for plastic response in which the wave speed for the current time step is fixed by the solution at the previous time step, and (3) for plastic response in which the wave speed is

determined iteratively by the solutions at the previous time step and at the current time step. Programming statements have been written for the first two levels of complexity.

**15427 APPLICATIONS OF BOUND AND MODE APPROXIMATION TECHNIQUES FOR LARGE DEFLECTIONS OF PULSE LOADED STRUCTURES**

Paul S. Symonds  
A. R. S. Ponter  
Brown University  
SL: AMMRC

Emphasis has been continued on developing an approach to estimates of major deflections due to impact or pulse loading in which advantage is taken of the simplicity of rigid-plastic treatment, but in which the main effects of elastic response are included. Hence no criterion for validity of a rigid-plastic treatment is required. The approach was shown to give extremely satisfactory agreement with experiments in cases where a rigid-plastic calculation fails to give useful results. In the cases treated the load pulse was very short (approximated well as impulsive). Difficulties with rigid-plastic treatments are still greater when the pulse is relatively long and the peak magnitude is only a few multiples of the plastic collapse load magnitude. A subsequent report reviewed the basic concepts of the method, with emphasis on the influence of pulse duration on the error of a rigid-plastic treatment. Illustrations were given of the failure of a rigid-plastic treatment to predict plastic deformations due to a long pulse, even though the ratio of plastic work to elastic energy capacity is large (for example, a 50 percent error when the energy ratio exceeds 10). Work has also been continued on the question of choosing the mode solution to obtain the best approximation, when several modes are available as in the case of impulsively loaded structures. Previous work led to suggested criteria by means of maximum values of lower bounds either on response time or on root mean square final deflection. A non-uniform (stepped) beam provides examples of a structure where several modes are available, and for which a criterion of selection is needed. A report comparing the suggested criteria is in preparation.

**Reports:**

No. 1-2 in previous edition.

**15513 LARGE AMPLITUDE COMPRESSION AND SHEAR WAVE PROPAGATION IN IMPACT-LOADED PMMA**

Y. M. Gupta  
D. D. Keough  
SRI International  
SL: AMMRC, BRL

Efforts have concentrated on obtaining accurate shear wave amplitude measurements at the impact surface and in the specimen interior. In addition, a method for measuring rear surface longitudinal particle velocity has been investigated in order to monitor in-material fractures. Experiments above and below 8 kbar compressive stress have been performed to obtain shear wave measurements at the impact surface and in the sample interior. Because of the nature of shear wave measurements, it is important to ensure that specimen assembly techniques do not influence the results. Experiments below 8 kbar and at angles of 8 degrees and 12 degrees show an elastic response. These results lend confidence to the specimen assembly techniques. Experiments at 17 degrees and greater angles show a rapid attenuation of the shear wave amplitude at the impact surface and at the interior gage locations due to localized failure at the interface. These data thus bound the shear wave amplitudes that can be coupled into the samples. Although these results are generally valid, there were instances when the amplitude decayed with wave propagation in experiments where elastic response was expected. In all such experiments, the front surface gage data showed interface slip. The exact cause of this slip has not been related unequivocally to any single experimental parameter. However, once the front surface slips, the shear wave amplitude is expected to decay with propagation. Above 8 kbar, the shear wave amplitudes are considerably smaller, and the amplitude decay is markedly rapid for shear deformations, which at lower compressive stresses propagate elastically. These observations support previous inferences about the strain softening observed in PMMA.

**Reports:**

1. Determination of the Impact Response of PMMA Using Combined Compression and Shear Loading, by Y. M. Gupta, *J Appl Phys.*

**15722 A STUDY OF TUBE BOUNDARY CONDITIONS AND OTHER SYSTEM PARAMETERS WHICH AFFECT GUN TUBE MOTIONS**

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SL: BWL, BRL SC: AMMRC, ARRADCOM

A recent survey of the open literature revealed only a few new papers on gun dynamics. Wear data were obtained on gun tubes and tube support sleeves. These data show that all dimensions are within production tolerances. A scale model analysis was performed to determine the scaling parameters for the model test weapon. Replica scaling will be used that gives correct scaling for all terms except those which contain gravity. Experiments will be performed using a scale model weapon that closely approximates the M68 105-mm Tank Gun. A caliber of 20 mm has been chosen that gives a scale of 1/5.25. The analytical model used at SwRI in previous studies of gun dynamics has been revised and converted to a new mini-computer. Improvements have been made in the routines that plot tube motions and that summarize and print results. Fabrication of the test fixture is in progress.

**15787 HIGH-STRAIN-RATE DISLOCATION MOTION**

W. G. Hoover  
University of California, at Davis  
SL: AMMRC, NLABS SC: AMMRC, BRL

A new method has been discovered for simulating adiabatic homogeneous deformations in microscopic computer simulations of many-body systems. The method uses a perturbed Hamiltonian, incorporating the product of the strain-rate tensor with Doll's Tensor, the coordinate-momentum product, summed over all particles. The perturbed Hamiltonian has many applications. Viscosities, yield strengths, nonequilibrium distribution functions, and theoretical approaches to these same functions have all been successfully treated. Computational effort has been devoted to (1) improved representation of defect-containing crystals for movies and (2) calculation of the properties of dislocations in the quasiharmonic approximation. Other efforts have resulted in being able to successfully minimize the energy of periodic crystals containing dislocation pairs, diagonalize

the force-constant matrix at the minimum, and compute the energy and entropy associated with these defect pairs.

**15868 FINITE-ELEMENT FORMULATION FOR THE ANALYSIS OF PLASTIC DEFORMATION OF RATE-SENSITIVE MATERIALS IN METAL FORMING**

Shiro Kobayashi  
University of California, at Berkeley  
SL: LCWSL

The formulation for a coupled analysis of viscoplastic deformation and heat transfer has been completed and the method was applied to the problems of solid cylinder and ring compression. The finite element formulation for the analysis of tube nosing at room temperature has been completed. The program was written and tested. The analysis was performed for steel tube nosing and the results are now being compared with the experiment in the report prepared by the Battelle Columbus Laboratories under the contract with the U. S. Army. The experiments of tube nosing at room temperature were conducted with aluminum and the results were compared with those by the finite element analysis. Comparisons were made in terms of load-displacement relationship and the detailed deformation, including geometrical changes of the workpiece. Simulation of nosing by the finite element method was good in solution accuracy and computation efficiency. For the analysis of tube nosing at elevated temperatures, literature on the subject was surveyed and the program for thermo-viscoplastic analysis was modified for the tube nosing problem. The program was tested and is ready for the analysis.

Reports:  
No. 1-2 in previous edition.

**15872 DYNAMICS OF COMPOSITE MATERIALS AND ELASTIC STRUCTURAL ELEMENTS WITH VARIABLE DIMENSIONS**

S. Nemat-Nasser  
Northwestern University  
SL: BWL SC: LANGLEY, AMES, MICOM

Work on the following item has been continued:  
Prediction of dynamic behavior of pretwisted

blades requires an accurate estimate of the shear center. This has also a bearing on curved structural elements and cables. An effort has been initiated to understand and estimate the location of shear center for pretwisted curved elements. In addition, the effect of anisotropy on dynamic behavior of composites has been studied and the following two papers have been completed and submitted for publication. (1) Harmonic Waves in Layered Transversely Isotropic Composites, in which the dispersive property of laminated elastic composites consisting of transversely isotropic layers is studied. Harmonic waves with various propagation directions with respect to the direction of layering are considered. Exact solutions are presented and compared with the results of the new quotient method recently developed by one of the authors. (2) Harmonic Waves in Fiber-Reinforced Orthotropic Elastic Composites, where the dispersive property of fiber-reinforced elastic composites consisting of orthotropic materials is studied. Based on a new quotient recently proposed by one of the authors, approximate dispersion curves are obtained, and the influence of anisotropy is illustrated.

*Reports:*

- No. 1-5 in previous editions.  
 6. Harmonic Waves in Layered Transversely Isotropic Composites, by S. Nemat-Nasser et al, *J Sound & Vibrn.*  
 7. Harmonic Waves in Fiber-Reinforced Orthotropic Elastic Composites, by S. Nemat-Nasser et al, *J Appl Mech.*

**15911 AN INTEGRATED APPROACH TO THE OPTIMIZATION OF HIGH-SPEED CAM-FOLLOWER SYSTEMS**

Ferdinand Freudenstein  
 Columbia University

SL: FCSCWSL SC: TARADCOM, LCWSL

The instrumentation of the high-speed cam-follower system (an automotive valve train for an overhead camshaft engine) was completed. The experiments involved the measurement of displacements, speeds, torques, forces and accelerations in the system over a range of speeds. This effort, which was extensive, has now been completed and was highly successful. Instrumentation and data acquisition functioned well. In addition to gathering pioneering experimental data, it is possible to sense and

observe the onset of toss and valve bounce - the prediction of which is essential to the design of such systems. Following the completion of the experimental runs, the research has concentrated on data reduction, interpretation and correlation with results obtained from the simulation model. Although this phase of the research is still continuing, creation of a predictive dynamic model appears possible. The development of such a model should be useful in the design of high-speed machinery, including ordnance equipment and automatic weapons.

*Reports:*

1. The Analytic Development and Experimental Verification of a Predictive Model of a High-Speed Cam-Follower System, by Albert P. Pisano, 1981, 284p, tables, PhD Thesis. (Volumes 1 and 2).

**15989 STUDIES ON THE FRACTURE MECHANISMS IN PARTIALLY PENETRATED FILAMENT REINFORCED LAMINATED PLATES**

K. Millsaps  
 R. L. Sierakowski  
 L. E. Malvern  
 University of Florida

SL: NLABS, BRL SC: AMMRC

A continuation of studies evaluating the principal fracture mechanisms in partially penetrated laminated composite plates systems fabricated from prepreg tapes has been pursued. Phase I has consisted of experimental studies involving the interaction of various impactor/plate configurations on extended delamination occurring within damaged specimens due to changes in impactor mass, velocity and plate geometry and material. Phase II of the program has consisted of flexural wave and delamination crack propagation studies. Details of the time history of the elastic waves in ballistically impacted glass/epoxy composite laminates have been obtained by using surface embedded strain gages. Phase III has related to assessing the residual strength and modeling of the damaged plates. Static three-point cylindrical bend tests have been conducted on both undamaged and damaged plates subjected to bending under a rounded-edge wedge centrally loading composite beam and plate specimens. Evaluation of test data for various cross ply and



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A. Solid Mechanics

III Engineering

angle ply damaged plates indicates that the retained static bending strength of such plates is within fifteen to twenty percent of the comparable loaded undamaged plate specimens, so long as the induced impact damage does not extend to the plate boundaries.

## Reports:

1. Transverse Cracks in Glass/Epoxy Cross-Ply Laminates Impacted by Projectiles, by N. Takeda et al, *J Mater Sci*.
2. Experimental Impactor/Plate Configuration Interaction Studies of Impacted Glass Fiber-Reinforced Composite Laminates, by Nobuo Takeda et al, *SAMPE Quart*.
3. Delamination Crack Propagation Studies of Ballistically Impacted Composite Laminates, by N. Takeda et al, *Expil Mech*.
4. Wave Propagation Experiments on Ballistically Impacted Composite Laminates, by Nobuo Takeda et al, *J Composite Mater*.
5. Studies of Impacted Glass Fiber-Reinforced Composite Laminates, by Nobuo Takeda et al, *SAMPE Quart* 12, (1981) AD A095 878
6. Microscopic Observations of Cross Sections of Impacted Composite Laminates, by N. Takeda et al, *Composites Tech Rev*.

## 16005 MECHANICS OF SURFACE DAMAGE IN ELASTOHYDRODYNAMIC CONTACTS

Herbert S. Cheng  
Leon M. Keer  
Northwestern University  
SC: MERADCOM

The main objective is to gain a basic understanding of the complex stress behavior governing pitting and scuffing failure in elastohydrodynamic contacts (EHD). The tasks include: (1) Two-dimensional contact stress analysis in EHD line contacts, (2) analysis of initial scuffing damage. In task 1, the parameter study of horizontal and vertical crack propagation due to asperity contact is completed and a paper to report these findings is in preparation. In task 2, the computer programming for the transient surface temperature calculation is completed. Its special case for uniform heating was compared with existing theories. Two-dimensional elastic solutions for the problem of frictional Hertzian contact on a half space surface and the problem of an angled crack embedded in a half space have been superposed to simulate by computer rolling contact fatigue. Cyclic loading

and unloading of a real surface crack embedded in a rolling wheel (caused by the crack passing through the rolling contact) is simulated by assuming that Hertzian stresses move across the cracked half space such that one passage is equivalent to one rolling cycle. By judiciously increasing the model's cracklength, an estimate of rolling contact fatigue life can be obtained using the rule.

## Reports:

1. Subsurface Cracking and Delamination, by L. M. Keer et al.

## 16036 THEORETICAL AND EXPERIMENTAL STUDIES IN THE RHEOMETRY OF VISCOELASTIC MATERIALS

Daniel D. Joseph  
Gordon S. Beavers  
University of Minnesota  
SL: CSL SC: CSL, BRL, AMMRC

Work has continued on a number of projects designed to produce information about the fundamental characteristics and behavior of viscoelastic fluids and solids. The projects which have been active during the period covered by this report, and upon which work is continuing, are summarized below. (1) *Rimming Flows*: The flow of a first order liquid coating the inside of a horizontal, steadily rotating cylinder has been studied analytically and experimentally. (2) *Eccentric Cylinders*: The theory for the slow steady motion of a viscoelastic fluid contained between eccentric rotating cylinders has been completed through second order using the theory of domain perturbations. (3) *Non-Newtonian Fluid Characterization*: An experimental program is in progress in which attempts are being made to relate molecular properties and solution concentrations of moderately concentrated polymer solutions to continuum characterizing parameters, and in particular to the climbing constant which is determined by means of rotating rod experiments. (4) *Rheometry of Viscoelastic Solids*: A general perturbation theory through second order has been developed for the deformation of traction-free surfaces which arise from perturbing the natural state of incompressible viscoelastic solids. The

theory has potential for rheological measurements on viscoelastic solids.

**Reports:**

- No. 1-5 in previous edition.
6. The Stick-slip Problem for a Round Jet, Part I, by S. A. Trogdon et al, *Rheol Acta*.
  7. The Stick-slip Problem for a Round Jet, Part II, by S. A. Trogdon et al, *Rheol Acta*.
  8. Instability of the Rest State of Fluids of Arbitrary Grade, by Daniel D. Joseph, *Arch Ratl Mech Anal*.
  9. Some Inequalities in the Theory of Simple Fluids, by Daniel D. Joseph, *Rheol Acta* 19, 389 (1980). AD A090 999
  10. Boundary Conditions for Thin Lubrication Layers, by Daniel D. Joseph, *Phys Fluids* 23, 2356 (1980). AD A095 235
  11. A Normal Stress Amplifier for the Second Normal Stress Difference by Leroy D. Sturges et al, *J Non-Newtonian Fluid Mech* 6, 325 (1980). AD A092 672
  12. Free Surface Problems Induced by Motions Perturbing the Natural State of Simple Solids, by P. M. Dixit et al, *Arch Ratl Mech Anal*.
  13. An Integral Invariant for Jets of Liquid into Air, by Daniel D. Joseph, *Arch Ratl Mech Anal* 74, 389 (1980). AD A095 244

# **16091 LIMITING PERFORMANCE OF NONLINEAR SYSTEMS WITH APPLICATIONS TO HELICOPTER VIBRATION CONTROL PROBLEMS**

Walter D. Pilkey  
University of Virginia  
SL: AMES, LANGLEY, EUSTIS

Antiresonance theory has been used to design dynamic vibration absorbers which reduce vibration at specified points on a sinusoidally loaded vibratory system to zero. In particular, absorber systems made up of springs and masses have been treated in detail. The sensitivity of the design to variations in system parameters has been examined. These analytical developments have been illustrated for a simply supported vibrating beam and for an elastic line helicopter model. This coupled fuselage/rotor elastic line model has been studied further by considering fuselage vibration as a function of its structural parameters. An efficient reanalysis technique has been used to evaluate the effects of modifying the structural properties of the helicopter. This reanalysis capability is exploited in the optimal design of vibration reduction devices by regarding them as variable substructures within the

helicopter. A local modification method which involves the attachment of a continuous structure, such as a beam or a plate, to the helicopter for vibration reduction has been developed. Currently work is in progress on active control systems and design for prescribed frequency response and active control systems. The latter will naturally lead to the application of methods of optimal control theory to the design of vibration reduction devices for helicopters. A demonstration model is being developed which uses a concept for reducing helicopter vibrations by sensing the unsteady lift forces on each blade, and by modifying the lift on the following blade accordingly.

## **16254 FUNDAMENTAL RESEARCH ON TRIBOLOGY**

Frederick F. Ling  
Rensselaer Polytechnic Institute  
SL: BWL, MERADCOM, EUSTIS, LEWIS,  
TECHNICAL SUPPORT CENTER

The research team has been engaged in capturing several research opportunities occasioned by new performance requirements of weapon systems, as well as those problem areas in tribology which are becoming tractable by virtue of new developments in instrumentation, computational capabilities and methods of analysis. Three areas, each designed to yield better information on the threshold of tribological failure are being studied. These are the role of surface topography on lubrication in the mixed film regime; the role of particulates on lubrication in the elastohydrodynamic regime; mathematical modeling of surface, surface layer and inter-surface phenomena, and associated computation relating to lubrication in the mixed film and elastohydrodynamic regimes. Another area has been concerned with the viscoelastic properties of low molecular weight polystyrene solutions in the high frequency regime. Other areas include (1) surface structure of operating bearing surfaces by speckle interferometry, (2) thermal effects in fluid film bearings, (3) the study of surface characterization and the kinetics of chemical reactions on surfaces and (4) an experimental and theoretical study of fluid flow and heat transfer in the contact line region (junction of a thin liquid film, vapor and substrate). Experimentally, various multicomponent systems have been evaluated on a preliminary basis.



## Reports:

1. A Systems Rationale for the Selection or Design of Tribological Surface Coatings, by Robert Johnson, pres Intl Conf on Met Coatings.
2. Preliminary Investigation into the Tribological Characteristics of Composite Coatings Sliding Against U.H.M.W. Polyethylene, by J. C. Roberts et al.
3. An Analytical Solution for Thermal Behavior of the Step Thrust Bearing, by J. A. Tichy et al, *J Lubrctn Tech* 102, 34 (1980).
4. Surface Mechanics of Layered Media, by Frederick F. Ling et al, *Proc Symp on Solid Contacts and Lubrctn*.
5. Wear Characteristics of Water-Lubricated Al<sub>2</sub>O<sub>3</sub>-Metal Sliding Couples, by Wolfgang Holzhauser et al, *Proc 3rd Intl Conf on Wear of Mater*.
6. The Viscoelastic Properties of Low Molecular Weight Polystyrene Solutions in the High Frequency Regime, by In-Young Koh et al, *Macromol*.
7. Abrasive Contaminants in Rolling Contact Bearings - A Review, by S. F. Murray et al.

# 16666 AN INVESTIGATION OF SOME BASIC PROPERTIES OF HYBRID COMPOSITE MATERIALS

T. W. Chou  
University of Delaware  
SL: AMMRC SC: BWL, AMES

The objective of the research is to develop analytical methods for predicting elastic moduli of hybrid composites and the compressive strength of unidirectional hybrids, and to conduct experiments to verify the above theoretical study. Theoretical predictions of the elastic moduli of multiphase short fiber composites have been made. The composites are treated as transversely isotropic. A theoretical analysis has been performed to understand the "hybrid effect" of the ultimate tensile strain of the hybrid composite, namely, the failure strain of a hybrid is different from those of composites reinforced with either of the "parent" fibers alone. A statistical analysis based upon the Monte Carlo Method has been adopted to simulate the complicated stochastic process of the fiber fracture in hybrids. Experiments have been performed on Kevlar/glass/phenolics hybrid systems. Intermingled hybrid composites have been successfully fabricated in the laboratory. Measurements are conducted on the effective elastic constants at six different relative fiber volume fractions. The total fiber volume fraction is fixed at 50%. The results are in good agreement with the theory.

# 16803 DIRECT AND INVERSE DIFFRACTION PROBLEMS FOR QNDE APPLICATIONS

J. D. Achenbach  
Northwestern University  
SL: BWL SC: LCWSL

In analytical work on the scattering of elastic waves by crack-like flaws, elastodynamic ray theory has been applied. Ray theory has the advantage of conceptual simplicity and intuitive appeal. A source produces a bundle of rays which propagate toward the flaw. The scattering of a bundle of rays by a flaw follows relatively simple rules which have been established by the study of canonical problems. At sufficiently high frequencies, diffraction at certain points on the crack edge, which have been called the "flash points", produces the dominant part of the scattered field. The flash points emit bundles of diffracted waves which propagate towards a point of observation. The basic theory has been presented in a number of papers. The application of elastodynamic ray theory to the scattering of bulk waves by surface-breaking cracks has been considered. Because of the presence of both a reflecting free surface and a crack, the number of possible ray paths is larger than for an interior crack. For L-L scattering by an interior crack generally only two rays need be considered. Mode conversion at the free surface and scattering by the interior edge of the surface-breaking crack may generate as many as nine relevant ray paths. The application of ray theory to the three-dimensional case of scattering of incident longitudinal waves by a surface-breaking crack, for the case that only the longitudinal signals are measured in the scattered field has been examined.

## Reports:

1. Direct and Inverse Methods for Scattering by Cracks in the High Frequency Range, by Jan D. Achenbach, *Trans 26th Conf of Army Mathematicians*.

# 16862 THE INFLUENCE OF SURFACE ROUGHNESS ON THE TRANSFER AND WEAR OF POLYMERS

N. S. Eiss, Jr.  
Virginia Polytechnic Institute and State University  
SL: LCWSL SC: BWL

The objective of the research is to determine the relationship between surface roughness and the

wear of polymers in multiple transversal, high speed sliding. During the reporting period the research has concentrated on two areas: (1) devising experiments to visualize the interaction of the polymer with asperities on a rough surface and (2) characterizing a surface by the peak density, peak curvature, and peak penetration and correlating these parameters with the results of wear experiments. One of the major unknowns in the wear of polymers on rough surfaces is the set of conditions which causes the formation of wear particles. Several techniques have been tried to visualize this process. A block-on-cylinder wear apparatus has been designed for use in the SEM. In these experiments a PVC cylinder is rotated against the surface of a steel block on which asperities have been made by grinding, or by scribing a groove on a polished surface. The phenomena which have been observed include the flow (plastic deformation) of rigid PVC over a contacting asperity without the formation of a wear particle, the buildup of PVC in the space between the polymer and the surface and the extrusion of this polymer out of this region in a direction perpendicular to the sliding motion, and the buildup of PVC which did not extrude but supported the load and minimized further transfer.

#### 17055 SHOCK WAVES IN THREE-DIMENSIONAL NONLINEAR ELASTIC SOLIDS

T. C. T. Ting  
University of Illinois at Chicago Circle  
SL: BRL SC: LCWSL, AMMRC

The transport equations for the amplitude of three-dimensional shock waves in nonlinear elastic fluids have been examined. It has been shown that, with the exception of the term which contains the mean curvature of the shock surface, the transport equations are almost identical to the transport equations for one-dimensional nonlinear elastic solids if one replaces the stress, strain and velocity in the latter by the pressure, specific volume and normal velocity, respectively. Therefore, the results obtained for one-dimensional shock waves regarding whether the amplitudes of jump in stress, strain and velocity grow or decay simultaneously can be applied here to the jump in pressure, specific volume and normal velocity. One of the objectives of the project was to find the propagation vector  $b$

along which the discontinuity in a physical quantity across the shock surface propagates. For elastic fluids, this propagation vector  $b$  is independent of the choice of the particular physical quantity on the shock surface. For nonlinear elastic solids, it was found that the vector  $b$  depends on the choice of the physical quantity on the shock surface.

#### Reports:

1. Intrinsic Description of Three-Dimensional Shock Waves in Nonlinear Elastic Fluids, by T. C. T. Ting, *Int'l J Eng Sci* 19, 629 (1981). AD A097 839
2. Characteristic Forms of Differential Equations for Wave Propagation in Nonlinear Media, by T. C. T. Ting.

#### 17064 THE EFFECT OF FLUID INERTIA AND VISCOELASTICITY IN SQUEEZE FILM DAMPER BEARINGS

John A. Tichy  
Rensselaer Polytechnic Institute  
SL: EUSTIS SC: TARADCOM

The research objective is to model and analyze squeeze film damper bearings for rotor dynamic systems where fluid inertia and viscoelastic effects create conditions under which the Reynolds equation is not valid. The work will consist of a combined analytical and experimental study. The analytical work will be the solving of a linearized set of equations for the fluid including fluid inertia, non-Newtonian fluid effects, and the appropriate geometry. The experimental research will have two parts: (1) rheological measurements to determine material parameters and (2) bench tests on a squeeze film damper rig to test the theory. The experimental apparatus has been completely designed and is currently being fabricated. The transducers have been obtained. The associated motor, pulleys, belting, etc., have been ordered. Two analytical papers have been completed. One is concerned with finding improved damper coefficients based on the small amplitude linear theory. The results are "exact" for the short bearing case, provided the amplitude of the shaft motion is small, which allows linear viscoelasticity theory to be used, and neglect of convective inertia terms. The other paper concerns an approximate technique which is applied to the case of arbitrary Reynolds numbers and arbitrary shaft motion for a Newtonian fluid.

**Reports:**

1. Effects of Fluid Inertia and Viscoelasticity on Squeeze Film Bearing Forces, by John A. Tichy, *ASLE Trans.*
2. An Approximate Analysis of Fluid Inertia Effects in Axisymmetric Laminar Squeeze Film Flow at Arbitrary Reynolds Number, by John A. Tichy, *Appl Sci Res.*
3. Effects of Fluid Inertia and Viscoelasticity on Squeeze Film Bearing Forces at Large Vibration Amplitudes, by John A. Tichy, *Wear.*

## 17067 EFFECT OF BLADE STRUCTURAL PARAMETERS ON HELICOPTER VIBRATIONAL CHARACTERISTICS

David A. Peters  
Washington University  
SL: AMES SC: AMES

Major efforts have been concerned with modifying computer programs in three ways. First, by adding additional structural-coupling parameters not included in previous work. Second, by streamlining the programs to give vibration and stability information with less computer time. Third, by modifying the pre- and post-processors so that the programs can be used by others. A fairly sophisticated blade-response program has been initiated to study the convergence of periodic shooting, of the automatic pilot, and of classical trim procedures.

## 17126 FATIGUE OF LAMINATED COMPOSITE STRUCTURES

George J. Dvorak  
University of Utah  
SL: AMMRC SC: BWL, AMES

Significant progress was achieved in analyzing the evolution of fatigue damage in metal matrix composites. It was established in past work that the major source of damage are long fatigue cracks which propagate in off-axis layers of composite laminates, in the directions parallel to the fibers. The stiffness reduction caused by these cracks can be as high as 50%. The analysis of the evolution process is based on single crack solutions in anisotropic solids and on the self-consistent method of averaging which accounts for the interaction between the cracks in each ply in an approximate way. This approach is advantageous

in the present case where one deals with several systems of parallel cracks in different directions. The material properties of the layers weakened by cracks of selected densities are then used in an evaluation of overall properties of the laminate, and of the stress states in individual plies. It was found that crack densities which correspond to cracks located within two or three fiber spacings apart can account for the maximum values of stiffness reduction observed in the experiments. Furthermore, the local stresses in zero-degree plies at overall loads corresponding to the endurance limit were found to be approximately equal to the endurance limits of unidirectional materials of the same composition. These results appear to verify an earlier conclusion that fatigue damage was caused by the long matrix cracks, and fatigue failure of a laminate by fatigue failure of the zero-degree plies. Further work is in progress which will make the technique a predictive one.

**Reports:**

- No. 1 in previous edition.
2. Finite Element Analysis of Elastic-Plastic Fibrous Composite Structures, by Yehia A. Bahei-El-Din et al, *Computers & Structures* 13, 321 (1981).

## 17216 MECHANICS OF PROJECTILE IMPACTS AGAINST THIN PLATES

Werner Goldsmith  
University of California at Berkeley  
SL: BRL, AMMRC SC: TARADCOM

The research objective is to extend the present state of theoretical modeling of the penetration of thin plates by projectiles including plate bulging, plastic deformation, and/or shattering. Theoretical and experimental studies will be carried out on steel and aluminum as target materials. These substances will be modeled by elastic-plastic-viscous constitutive equations noting their yield limits and by Hugoniot relations in the high pressure regime. The studies will include membrane and bending effects. Large deformations and strains will be considered. Adiabatic conditions will be assumed. In the experimental part, both pneumatic and powder guns will be used. The entire history will be photographed and analyzed. Approximately 100 penetration and perforation tests have been made on aluminum plates. A force gage embedded in projectiles has been developed and is capable of

measuring the force history during contact of the projectile with the plate. A velocity gage developed in conjunction with a rock penetration project has been adapted for the present series of tests. This has resulted in force-velocity curves for the projectile where it was found that just at the point of perforation, the peak force exerted by the projectile drops substantially. A simple analytical development considering a lumped parameter system consisting solely of viscous and inertial forces provides amazingly good correlation with the experimental findings.

#### 17221 PLASTIC DEFORMATION AND FRACTURE OF STEEL USING DYNAMIC LOADING

J. Duffy  
Brown University  
SL: AMMRC, BRL

The research objective is to conduct experimental investigations of the temperature and rate effects in dynamic plasticity and dynamic fracture. Specifically, the research is to determine the conditions required for shear band formation and fracture toughness at high rates of loading. Torsional Kolsky bars and IF sensors will be used to conduct real time measurements of temperature, strain, and strain rate during the formation of shear bands. The results will be analyzed by the constitutive relations developed in earlier projects. For the toughness measurement, explosively loaded bars will be employed over a wide range of temperatures. The latter program will include fracture initiation studies of heat treated steels. In previous experiments with two low carbon steels (AISI 1018 cold-rolled and 1020 hot-rolled) the temperature ranges for cleavage fracture under quasi-static and dynamic loading were established. Microscopic examination of the specimens after testing showed that the fractures on the lower shelf initiate at carbide plates that precipitate along the ferrite grain boundaries, and that this result holds true for both quasi-static loading and dynamic loading. In order to pursue this subject further, an investigation was started to determine the fracture behavior of similar steels with fewer and smaller carbide plates.

#### Reports:

1. The Size and Density of Intergranular Carbide Plates in 1020 HRS and Their Role During Brittle Fracture Initiation: A Preliminary Report, by J. C. Lambropoulos et al, Jan 81, 22p, figures, TR. AD A097 022

#### 17357 HOLOGRAPHIC DISPLACEMENT ANALYSIS WITH FIBER OPTICS

John A. Gilbert  
University of Wisconsin at Milwaukee  
SL: BRL, LCWSL SC: LEWIS, BWL, AMMRC

The research objective is to develop novel non-contact, non-destructive methods for measuring displacement and/or strain at ordinarily inaccessible locations. Fiber optics will be used to gain access to remote area of structures. The displacement and strain will be determined by constructing laser produced, image plane holograms. Tests carried out immediately prior to the official start of the project, concentrated on incoherent and coherent bundles made up from individual fibers of relatively large diameter. A subsequent review of literature on fiber optics, Task 1, has not only provided valuable information on which to base current studies, but has uncovered investigations which will provide the basis for Phase II; namely, to develop a theory which governs holographic displacement patterns generated with fiber optics. Research carried out has shown that fringe contrast can be significantly improved by imaging the end of the fiber bundle used for illumination onto the test surface.

#### Reports:

1. Remote Displacement Analysis Using Multimode Fiber Optic Bundles, by John A. Gilbert et al, *Exptl Mech.*

#### 17575 OPTIMUM DESIGN AND AUTOMATED DYNAMIC ANALYSIS OF FLEXIBLE MECHANISMS

Iradj G. Tadjbakhsh  
Rensselaer Polytechnic Institute  
SC: TARADCOM, AMMRC, LCWSL

Research objectives will include: (1) Develop algorithms for optimum design for mechanisms with applications. (2) Develop an automated derivation of equations of motion of multiple flexible body mechanisms. (3) Investigate dynamic



instability, vibration and resonance in flexible mechanisms. (4) Develop and apply modal analysis and finite element formalism to linearized infinite degree of freedom mechanism dynamics. The initial approach will be analytical and will be devoted to developing an automated derivation of the equations of motion of three dimensional flexible linkages after which appropriate numerical methods will be chosen for solutions. A systematic development of the nonlinear equations of motion of a planar system of flexible elastic linkages has been carried out. The equations, which take into account the important effect of rotatory inertia for high speed mechanisms, have been specialized for the case of small deformations. Equivalent variational formulations have also been determined. Using Galerkin procedure, a rapidly convergent series solution is obtainable for the case of links of constant cross-section. Moreover, for certain geometries, the equations of motion can be integrated in closed form. These closed form solutions provide a valuable check on the numerical calculations involved in the general cases.

*Reports:*

1. Stability of Motion of Elastic Planar Linkages with Application to Slider Crank Mechanism, by I. G. Tadjbakhsh, *ASME J of Mech Design*.
2. Dynamic Instability of the Elastic Coupler of a Four-Bar Mechanism, by M. C. Constantinou et al, *ASME J Eng for Industry*.

**17647 AN INVESTIGATION OF INHOMOGENEOUS INELASTIC DEFORMATION THROUGH PLATE IMPACT RECOVERY EXPERIMENTS AND MATHEMATICAL MODELING OF PROPAGATING LOCALIZED DEFORMATIONS**

Rodney J. Clifton  
Brown University  
SI: AMMRC, BRI

The research objective is to conduct both theoretical and experimental studies of heterogeneous deformation in solids at high rates of loading. The research is focused upon the kinetics of the development of shear bands, twins, and martensite plates. Short duration shock pulses will be used to conduct recovery experiments on the

microdynamics of the heterogeneous deformation. Step-by-step development of the localization will be observed by an optical and an electron microscope as well as transmission electron microscopy. Analysis will be based upon his previously developed model of general instability phenomena associated with high rates of deformation.

**17689 THE ANALYSIS OF ELASTIC-PLASTIC DEFORMATION AND STRESS AT FINITE STRAIN AND THEIR EVALUATION**

Erastus H. Lee  
Stanford University  
SI: BWL, AMMRC SC: FCSCWSL

The anomaly in finite strain elastic-plastic theory pointed out and solved by the principal investigator in the course of his recent ARO contract will be generalized from isotropic hardening to more general material behavior. This will necessitate further development of the present finite-element computer program. Also, it is planned to develop a technique for dealing with the onset of contact between tool and workpiece where the point of contact is not known *a priori*. The equations of motion, continuity and constitutive relations will be set up. Bauschinger effect and anisotropy will be included. The solution will be developed in terms of deformation gradients.

**17731 KINEMATIC AND DYNAMIC ANALYSES OF HIGH-SPEED INTERMITTENT-MOTION MECHANISMS**

T. W. Lee  
Rutgers University  
SI: BWL SC: ICWSL

The research objective is to develop theory and computational procedures for the kinematic and dynamic analyses of high-speed intermittent mechanisms. Analytic methods will be applied to selected mechanisms to formulate a dynamic model of impact and response under various operating conditions. The experimental part involves measurements of kinematic and dynamic response, stresses, wear, and fatigue.

### 17758 INTERACTION AMONG INHOMOGENEITIES AND INCLUSION

Toshio Mura  
Northwestern University  
SL: LCWSL SC: BRL, AMMRC

To investigate mechanical behavior of inhomogeneous materials under given applied loads. The elastic field due to an ellipsoidal inclusion which has an arbitrarily distributed eigenstrain will be determined as well as the field for the interaction among two or more ellipsoidal inhomogeneities. This information provides the basis for describing interaction among inhomogeneities by simulation of inclusion problems with properly chosen eigenstrains, termed the equivalent inclusion method.

### 17830 STOCHASTIC STABILITY RESPONSE OF MULTIBLADED ROTORS

J. E. Prussing  
Y. K. Lin  
University of Illinois at Urbana  
SC: AMES, LANGLEY

The research objective is to obtain realistic stability boundaries and response characteristics for multibladed rotors operating in the presence of turbulence. A theoretical analysis will be carried out based on linearized equations of motion and the method of stochastic averaging.

### 18447 VISCOELASTIC AND DYNAMIC PROPERTIES OF ARMOR MATERIALS UNDER HYPER- VELOCITY PENETRATION

C. A. Broniarek  
Tuskegee Institute

The objective of the research is to study the hypervelocity mechanism of armor penetration by a projectile of dissimilar material. The problem will be attacked both experimentally and analytically. The target and projectile will be described through a generalized integro-differential description accounting for elastic and inelastic properties as

well as thermal and strain rate effects. Emphasis will be placed on scale modeling.

### B. FLUID DYNAMICS

### 14254 STUDIES OF SLENDER, AXIALLY SYMMETRIC BODIES AT INCIDENCE AT HIGH REYNOLDS NUMBERS

David S. Dolling  
Seymour M. Bogdonoff  
Princeton University  
SL: BRL, MICOM

An experimental investigation is being made of the effects of nose blunting on the flow over a tangent ogive cylinder model. Seven different nose tips have been tested. The first set of measurements made included axial and circumferential pressure distributions and boundary layer pitot pressure surveys at different axial stations on the model wind and leesides. Some additional experiments have been carried out. A new model has been built, with the 3 caliber ogive nose instrumented with pressure tapings. This nose could be fitted with either a flat faced or hemispherical tip, both of which were also instrumented with pressure tapings. Comparisons have been made between measured boundary layer velocity profiles on the pointed tip model and predictions from Dr. Walter Sturek's codes. Analysis of the boundary layer profiles measured using the different blunted nose tips is still in progress. This will be fully detailed in the final report.

### 14741 UNSTEADY FLOW OF AN OSCILLATING AIRFOIL IN TRANSITIONAL MOTION

C. M. Ho  
University of Southern California  
SL: AMES

*The Mean Streamwise Velocity:* Two types of wake were identified from the mean streamwise velocity profile. The two boundary layers merge into a bell shaped viscous wake which is relatively narrow. The inviscid wake extends about one chord length above and below the trailing edge. The velocity defect is about 10% of the maximum velocity defect. However, a substantial amount of

momentum defect, i.e. drag, is contributed by the inviscid wake due to the large width. Unfortunately, the inviscid wake is neglected in experimental literature. *The Mean Transverse Velocity:* Accurate measurements of the mean transverse velocity were made. The data were checked by integrating the transverse velocity across the wake at several downstream stations. *The Self Similarity:* In the near wake of the unsteady airfoil, the mean velocity profiles, the fluctuating velocity profiles and the Reynolds stress profiles are normalized to the mean velocity defect. Within one chord length downstream from the trailing edge, only the mean streamwise velocity reaches the self-similarity. *The Phase Average Velocity Profiles:* The phase averaged streamwise and transverse velocity profiles reveal the unsteady wake at each instant during the oscillating cycle. At 5 degrees mean angle of attack and 0.54 reduced frequency, the phase averaged viscous wake did not change its shape appreciably because there was no unsteady separation in the operating conditions. The velocity profile in the inviscid wake changes at each phase angle. This variation might be due to the alternately shed vortices. The phase averaged vorticity contours could provide the clue.

#### Reports:

- No. 1 in previous edition.
2. Unsteady Kutta Condition of a Plunging Airfoil, by Chih-Ming Ho et al.

#### 14990 AN EXPERIMENTAL STUDY ON INERTIAL OSCILLATIONS IN A ROTATING CYLINDER DURING SPIN-UP OF THE FLUID FROM REST

Keith Aldridge  
York University  
SI: BRL

Work has been completed on the complex eigenfrequency for the (1,2,1) inertial wave in a fluid cylinder during spin-up of the fluid from rest. The wave is excited during spin-up by a precessing lid with half amplitude  $\epsilon$  radians. Both eigenfrequency and decay rate are determined from inversion of disturbance pressure data recorded during ring down of the oscillation. Near resonance

during spin-up, the precessing lid is switched off so that the fluid oscillation then decays at its natural frequency appropriate to that point in time for spin-up of the fluid. Work has also been completed on the onset of a wave instability of the mean azimuthal flow during spin-up from rest. Near the (1,2,1) resonance the instability exists during spin-up and as expected occurs earlier in time for larger tilts of the precessing lid which excites the inertial wave. Experiments have been extended to study the time dependence of the complex eigenfrequency for the (1,1,1) inertial wave during spin-up from rest. Close proximity in eigenfrequency to the (3,3,1) and (2,2,1) modes leads to significant interference from these modes. Furthermore the onset of the wave instability occurs at much smaller perturbation near the (1,1,1) mode than in the case of the (1,2,1) mode. Recovery of the decay rate and eigenfrequency for the (1,1,1) mode during spin-up was complicated by these two factors. The decay rate during spin-up is greater than its steady state value so that qualitatively this is in agreement with all previous experiments.

#### Reports:

1. Inertial Waves in a Fluid Partially Filling Cylindrical Cavity During Spin-Up From Rest, by S. Stergiopoulos et al, *Geophys and Astrophys Fluid Dynamics*.

#### 15336 VISUALIZATION OF THE BOUNDARY LAYERS OF SPINNING AND NON-SPINNING AXISYMMETRIC BODIES

T. J. Mueller  
R. C. Nelson  
University of Notre Dame  
SI: BRL SC: ARRADCOM

Most of the effort was devoted to obtaining flow visualization data to determine the influence of nose bluntness and the influence of sound for the sharp secant ogive nose configuration. A large number of still photographs was taken of the baseline model (i.e., sharp secant ogive nose) and the same model with the nose section replaced with 10%, 20% and 30% spherically blunted noses. The Reynolds numbers and spin rates used were determined from previous experience, and the angle of attack was held at zero. For a Reynolds

number  $Re_i = 1,030,000$  and zero spin rate, there is a marked difference in the Tollmien-Schlichting wave and truss (i.e., vortex loops) location and formation between the sharp nose and the 10% blunt nose. In going from the 10% to the 20% blunt nose, all other conditions remaining the same, the Tollmien-Schlichting waves form in about the same location but are much better defined for the 20% nose; also, there are fewer rows of trusses (i.e., one or two) before complete breakdown to turbulence. Increasing the bluntness from 20% to 30% does not seem to affect the Tollmien-Schlichting waves or the trusses very much. At  $Re_i = 928,000$  and a spin rate of 1500 rpm, the Tollmien-Schlichting waves which are barely visible for the sharp nose case become very clearly visible with the 10% nose bluntness. In going from 10% to 20% nose bluntness, very little change is evident in the individual events except that they all occur further upstream for the 20% nose. For the 30% blunt nose at these conditions, the striations break down occurs about two-thirds of the distance along the mid-section. The simultaneous occurrence of Tollmien-Schlichting waves and cross-flow vortices observed in this investigation has generated considerable interest in the fluid dynamics community.

#### 15657 BASIC TECHNOLOGY OF SQUEEZE-FILM DAMPERS FOR ROTOR-DYNAMICS CONTROL

Coda H. T. Pan  
Shaker Research Corporation  
SC: TARADCOM, LEWIS

Short bearing theory analysis at the submerged condition established that the damper coefficients depend on both the size of the whirling orbit and the condition of cavitation incipience, which is an indicator of the lowers film pressure. With pressurization of the lubricant supply at one end, the cavitation incipience condition becomes also dependent on the supply pressure; while friction and flow coefficients are universal functions, the cavitation pattern and damper coefficients are also dependent on the degree of pressurization. Further analysis showed that the damper coefficients can be expressed as explicit functions of the cavitation

offset parameter which represents the degree of pressurization. Consequently, a full set of results are given by two parameter universal functions while the cavitation offset parameter is separately determined according to the degree of pressurization. Experimental studies were performed on a test rig featuring a damper of dimensions (length = 4 cm, diameter = 8 cm) with a radial gap of 0.08 mm. A dynamic load up to 155 gm-cm can be obtained by inserting screws into holes on a rotating disk at a radial distance of 3.81 cm from the axis of rotation. Squeeze motion of the damper was sensed by non-contact displacement proximity probes. The outer housing is transparent, so that the cavitation pattern can be viewed either under continuous illumination or under synchronized stroboscopic lighting. Tests were performed up to 5,400 rpm. Observation with synchronized stroboscopic lighting revealed a coherent overall pattern in the cavitation domain suggesting a nearly stationary condition relative to the dynamic load.

Reports:  
No. 1-3 in previous edition.

#### 15710 A STUDY OF THE AERODYNAMIC INTERACTIONS OF THE TAIL ROTOR AND FIN

Philip F. Sheridan  
Boeing Vertol Company  
SL: EUSTIS SC: LANGLEY

Work has been concerned with the interactions affecting the fin in cruise flight with special reference to the case of tail rotor loss where the fin must provide directional trim to the helicopter. The findings are as follows: (1) Adverse fin loading due to tail rotor presence is significant at all forward speeds, (2) Effectiveness of the fin as a stabilizer is reduced in a nonlinear manner by the flow generated by the rotating inner rotor i.e. hub, pitch links, blade shanks. This effect persists and is altered only slightly with the full rotor and tail rotor present, (3) This nonlinear shape of the fin yawing moment variation with sideslip causes a serious degradation to aircraft directional stability at left sideslip. In the important tail-rotor-off case, the



model data indicated neutral static stability over a sideslip angle range from -25 degrees to +5 degrees, and (4) This characteristic seriously compromises the trimability of the helicopter in the emergency tail-rotor-out condition. Also the loss in effectiveness makes static equilibrium about the yaw axis (vs. the rotor torque) possible only at a high descent rate. Thus the flow at the empennage evidently is not understood for either the forward flight case above or the hover-in-a-wind case studied earlier. To remedy this basic deficiency in the data base, a third year program has been developed for flow visualization and measurement in the wind tunnel at significant operating conditions.

*Reports:*

No. 1 in previous edition.

## 15743 DYNAMIC COUPLING OF BOUNDARY-FITTED COORDINATE SYSTEM WITH THE NAVIER-STOKES EQUATION

Joe F. Thompson  
Carl Davis  
Mississippi State University  
SL: BRL, AMES, MICOM

The development of the new code incorporating the dynamic coupling of the coordinate system with the Navier-Stokes solution has been slowed considerably by lack of personnel. Development of the concepts of the means by which the flow solution exerts its influence on the coordinate system has continued.

## 15780 AN EXPERIMENTAL INVESTIGATION OF UNSTEADY TURBULENT BOUNDARY LAYERS

B. R. Ramaprian  
V. C. Patel  
University of Iowa  
SL: AMES, LANGLEY

A large part of the activity continues to be the fabrication and assembly of the unsteady flow

water tunnel. All the sections constituting the tunnel have been fabricated and galvanized/plated. On the instrumentation and data acquisition side, further software development has been made on the HP1000 data acquisition system. Programs have been developed and tested for obtaining smoothed power spectra and autocorrelations for unsteady turbulent flows. A novel calibration technique has been developed for the measurement of wall shear stress in unsteady turbulent flows using a flush wall mounted heat flux gauge. Along with the above work, investigation of periodic oscillatory pipe flow has continued. Preliminary but substantial data have been now obtained in oscillatory flow of water in a pipe of 50 cm dia at a mean flow Reynolds number of 50,000, and oscillation frequency of 3.6 Hz, and an oscillation amplitude of about 10%. The large amount of data already generated are on computer file and are presently being analyzed, for information on the turbulent structure. On the computational side, a time dependent finite difference method is being developed for the calculation of unsteady turbulent boundary layers in arbitrary temporal and spatial pressure gradients. The numerical scheme being developed will allow alternate turbulence models to be studied.

*Reports:*

No. 1-2 in previous edition.

## 15866 THE DEVELOPMENT OF A SEPARATION MODEL FOR TWO- DIMENSIONAL AIRFOILS IN TRANSONIC FLOW

Frank A. Dvorak  
Analytical Methods, Inc.  
SL: EUSTIS, LANGLEY SC: AMES

The objective of this research effort is the development of an analysis method for transonic flow over two-dimensional airfoils having regions of separated flow. The present work is based on a separation model developed by Analytical Methods. Jameson's full potential finite-difference method was chosen for the transonic case. The major effort has been the extension of the method to the higher Mach number regime. Considerable

numerical difficulties were encountered as Mach number was increased, in particular, coarse grid solutions would not converge at the higher Mach numbers. Several different approaches were investigated with no success, until it was determined that a combination of the coarse grid and Jameson's Poisson solver were causing the numerical problems. A new solution procedure has been developed using a combination of multi-grids and, initially, a relaxation technique. After approximately 50 iteration cycles on the dense grid, the poisson solver can be utilized to accelerate convergence. Solutions have now been generated for angles of attack up to 14 degrees at Mach numbers of 0.5 and 0.6, in good agreement with experiment. A more comprehensive comparison with experiment is now under way.

*Reports:*

1. The Development of a Separation Model for Two-Dimensional Airfoils in Transonic Flow, by Frank A. Dvorak, pres AIAA 14th Fluid and Plasma Dynamics Conf.

**15905 DEVELOP A NAVIER-STOKES ANALYSIS FOR THE AFT-END FLOW FIELD OF AERODYNAMIC VEHICLES**

Henry McDonald  
Scientific Research Associates, Inc.  
SL: BRL, MICOM SC: EUSTIS ARRADCOM

One effort has concentrated upon extending the Navier-Stokes calculation procedure (MINT code) used to calculate the jet plume interaction problem so as to allow for the use of time-dependent nonorthogonal coordinate systems in future computations. With this goal in mind the version of the MINT code which had been used to calculate the backstep problem has been combined with a version of the MINT code which had been developed for general time-dependent coordinates. The resulting code contains the boundary conditions, corner point logic, initial profile generation routine, etc. which are required for the back step type problems with a time-dependent coordinate system code. Another effort has focused upon developing a simple adaptive grid for a transonic shock boundary layer interaction. This simple adaptive grid automatically creates a fine

mesh at the transonic shock location. A calculation with this grid is being made for flow conditions corresponding to the data of Mateer, Brosh and Viegas, in which a transonic shock occurs within a circular duct. The adaptive mesh calculation is operational and the calculation is currently being assessed with effort focusing upon the turbulence model.

**16030 ACQUISITION OF A SYSTEMATIC DATA BASE FOR WING ALONE CHARACTERISTICS AT HIGH ANGLES OF ATTACK**

J. N. Nielsen  
Nielsen Engineering & Research Inc.  
SL: MICOM

Because the 6x6 ft. tunnel at Ames Research Center has been shut down for several months, little work has been done. In July, a trip was made to Langley to discuss the interface with their tunnel. A concern was expressed about possible sting interference effects between the shroud (used with force wings only) and the windward surface of the wings at high angle of attack. A dummy shroud is being built to be mounted on the pressure wing-sting and plans are being made to run tests with and without the dummy shroud to see if the effects are significant. If the effects are significant, the options of changing the shroud configuration or applying a correction to the data for the sting effect will be considered.

**16061 AN EXPERIMENTAL AND ANALYTICAL STUDY OF HELICOPTER AERODYNAMICS IN GROUND EFFECT**

H. C. Curtiss, Jr.  
Princeton University  
SL: AMES, EUSTIS

The experimental data taken at three height-to-diameter ratios at advance ratios from 0 to 0.1 has been reduced and is being analyzed and compared with a theoretical model. The theoretical model consists of a doublet string model similar to that developed by Heyson to represent the rotor wake.

The ground vortex is modeled by a line vortex. The location of the ground vortex as a function of rotor flight conditions and height-to-diameter ratio was determined experimentally from the flow visualization studies. Ground vortex strength was determined empirically in two ways: (1) by determining the value of the strength necessary to match the pitching and rolling moment data and (2) determining the value of vortex strength such that the ground vortex and its image would be stationary in the freestream flow. The pitching and rolling moments and thrust and torque predicted by these two approaches were compared with experiment. The first method gives, in most test conditions, the strongest vortex. The strength required to predict the moment variations is too large when used to calculate the thrust and torque variations. The second approach (2) appears at this time to give a good estimation of the thrust and torque variations but is too small to predict the hub moment variations. It appears that the lack of correlation in the moments in this approach is due to the sensitivity of the moment prediction to knowledge of precise locations of the ground vortex. Efforts are continuing on the theoretical development to resolve these issues. The theory indicates that the ground vortex strength is one to two orders of magnitude larger than the blade tip vortex.

#### 16101 AN INVESTIGATION OF THE AERODYNAMICS OF NON-AXISYMMETRIC PROJECTILES

Victor Zakkay  
General Applied Science Laboratory, Inc.  
SL: MICOM. BRL SC: ARRADCOM

An experimental investigation in the use of non-axisymmetric boattails for drag reduction of projectiles is being performed at  $M_\infty = 6$ . Previous investigations at lower Mach numbers have shown that desirable drag reduction can be obtained through the use of non-axisymmetric boattails. In particular, this research considers boattails of circular, triangular and square base cross-sections compared to the results of a projectile having an aft portion composed of a straight circular cylinder. The research has concentrated on the following

problems: (1) Tests performed on the square boattail. (2) Heat transfer instrumentation on the conical and triangular boattail. (3) Comparison of the pressure distribution data with inviscid and viscous codes. (4) A modified analytical scheme for the prediction of body forces for the various configurations.

#### Reports:

1. Analytical and Experimental Investigation of a Boattail Projectile at  $M_\infty = 6$ , by V. Zakkay et al.

#### 16280 BALLISTICS OF SHELLS CONTAINING A VISCOELASTIC FLUID

R. S. Rivlin  
Lehigh University  
SL: BRL SC: LCWSL CSL

Work has been continued on the run-up problem for a viscoelastic fluid in the following configurations: (1) The fluid is contained between two infinite parallel plates which are suddenly set in rectilinear motion, with constant velocity, parallel to themselves, (2) The fluid is contained in an infinite cylinder which is suddenly set in motion with constant velocity, in the longitudinal direction, and (3) The fluid is contained in an infinite cylinder which is suddenly set in uniform rotational motion, with constant velocity, about the cylinder axis. Solutions have been obtained by using a Laplace transform procedure and solving the resulting equations. However, an alternative approach has been found which could be taken, and, particularly from a physical point of view, gives a better insight into the way in which the run-up takes place. This is essentially a ray-optical approach which follows the propagation of a wave-front from the boundary through the first few reflections at the axis and boundary in cases (2) and (3) when the container is cylindrical. The results obtained complement and confirm those previously obtained.

#### Reports:

1. Run-Up and Spin-Up in a Viscoelastic Fluid. I, by J. V. Kazakia et al, *Rheol. Acta*.

# 16294 DEVELOPMENT OF A GENERAL METHOD FOR THE FREE-WAKE ANALYSIS OF HELICOPTERS IN HOVER AND FORWARD FLIGHT

Luigi Morino  
Boston University  
SL: AMES

The objective of the first-year effort was to develop a program to obtain the steady-state geometry of the steady-state wake for a rotor in hover. A pilot computer code was assembled in order to study the feasibility of the proposed method. The test involved comparison with existing experimental and theoretical results. This pilot code uses a pseudo-time-domain analysis to obtain the desired steady-state solution. The program has been debugged from a grammar point of view, and the debugging from the algorithm point of view is now underway. This algorithm-debugging process can be divided into three separate areas: first, the portion of the program that performs pseudo-time-domain analysis with a prescribed wake; second, the portion of the program which calculates the velocity of the points of wake; and third, the portion of the program which calculates the new wake. The second-year effort calls for an actual time-domain (instead of pseudo-time-domain) analysis. A new program is being written which is much more modular, and utilizes a frame of reference moving with the wake. This will be beneficial, as the program will be much easier to use and debug. In addition, the new frame of reference would appear to be the correct formulation for future use, as it would give a more accurate account of a complex wake. Also, it was found that the fixed frame of reference was too cumbersome. These modifications should help the investigation of the instability problem.

# 16595 CURVATURE EFFECT ON EFFECTIVENESS AND HEAT TRANSFER IN THREE-DIMENSIONAL FILM COOLING

E. R. G. Eckert  
R. J. Goldstein  
University of Minnesota  
SL: BRL, LEWIS SC: TARADCOM

Four different studies related to film cooling are in various stages of completion. These include

research on: (1) the effect of flow condition (laminar versus turbulent) on film cooling; (2) film cooling with two rows of holes on gas turbine blades; (3) mass transfer on a circular cylinder; and (4) flow and film cooling in a curved wind tunnel. The influence of a laminar versus a turbulent boundary layer and laminar versus turbulent film cooling injectant flow on film cooling performance has been studied. The experimental program is essentially completed and the results are being analyzed for final publication. A thesis is being completed on film cooling with two rows of holes in the gas turbine cascade. Effects similar to those found for a single row are observed but with a much less pronounced influence as had been predicted. A naphthalene cylinder has been constructed for a study of the mass transfer, and by analogy the heat transfer on a cylinder in cross-flow. In order to analyze more fully the influence of curvature on film cooling, a special small tunnel was designed and is under construction. The tunnel will have constant curvature on both the outside (concave) and inside (convex) surfaces. Injection systems with various diameter holes will be used to control the ratio of hole diameter-to-surface radius.

## Reports:

1. Film Cooling in a Plane Turbine Cascade, by R. J. Goldstein et al. *Heat Transfer in Gas Turbines*, ed by A. Wood, Hemisphere Press (draft chapter).
2. Heat Transfer in High-Temperature Gas Turbines: Film Cooling and Impingement Cooling, by R. J. Goldstein, *Proc 80 Joint US-Japan Heat Transfer Seminar*.
3. The Influence of a Laminar Boundary Layer and Laminar Injection on Film Cooling Performance, by R. J. Goldstein et al.

# 16651 RESEARCH ON WALL TURBULENCE

Ron F. Blackwelder  
John Laufer  
University of Southern California  
SL: AMES SC: BRL

Research has concentrated on studying the breakdown of the counter-rotating streamwise vortex system in the wall region of turbulent boundary layers. The close similarity between the bursting phenomenon and the initial breakdown of bounded laminar flow into turbulence was also explored. A single heated roughness element was placed in an unstable laminar boundary layer. The turbulent temperature distribution behind the



wedge spread as a classical wake having a spread half-angle of approximately 1 degree. However, the turbulent wedge of fluid behind the roughness element spread with a corresponding angle of 9 degrees as determined by the mean and fluctuating velocity traverses. Evidently the initial turbulence as marked by the heated fluid created sufficiently large disturbances that it destabilized the laminar boundary layer causing it to breakdown into turbulence. This is in contrast to the classical entrainment mechanism by which turbulence is usually acquired from steady flow fields. An experiment is being prepared to investigate the behavior of the bursts as they leave the viscous region and migrate into the logarithmic region near the wall. Some exploratory tests were made with thermocouples manufactured from extremely small wires in the wall region of a slightly heated boundary layer. The thermocouples are sufficiently small that they have adequate time response to follow the turbulent fluctuations and have a sampling volume significantly less than hot-wires.

### Reports:

1. On the Growth of Turbulent Regions in Laminar Boundary Layers, by Mohamed Gad-el-Hak et al, *J Fluid Mech.*

## 16719 FLUID DYNAMIC MECHANISMS AND INTERACTIONS WITHIN SEPARATED FLOWS AND THEIR EFFECTS ON MISSILE AERODYNAMICS

A. L. Addy  
W. L. Chow  
H. H. Korst  
R. A. White  
University of Illinois, at Urbana  
SI: BRL, MICOM SC: LCWSL

Flow separation experiments are currently being conducted to establish the relationship between the initial shear layer properties and the corresponding boundary layer values as a function of the freestream Mach number, separation point process, and local geometry at separation. Comparison of these data to available predictive methods and correlations with shear layer parameters are in progress. Experimental hardware is being designed and fabricated to investigate a group of separated flow problems through detailed LDA and pressure measurements. Experiments

currently being considered are: (1) formation and development of a free shear layer, (2) recompression of a free shear layer, (3) recirculation in a base flow region, and (4) recompression of two impinging free shear layers. All of these experiments are planned for two-dimensional, supersonic flows. The theoretical investigation of the jet-slipstream interaction within the transonic flow regime was continued. Specifically, base pressure solutions have been found for such problems with a simplified model for the recompression process and restricted flow conditions. The effect of nozzle pressure ratio, flight Mach number, nozzle diameter-to-base ratio can be explored within a limited range of flight conditions. The numerical solution of the Navier-Stokes equations for flow over an airfoil of arbitrary configuration is under investigation. The turbulent flow process is modeled through the consideration of the turbulent kinetic energy and its dissipation.

### Reports:

- No. 1 in previous edition.
2. TRANNOZ: A Computer Program for Analysis of Transonic Throat Flow in Axisymmetric, Planar, and Annular Supersonic Nozzles, by J. C. Dutton et al, Apr 80, 89p, tables, TR. AD A091 924
3. Transonic Flow in the Throat Region of Axisymmetric Nozzles, by J. C. Dutton et al, *AIAA J* 19, 801 (1981).

## 16760 CONTINUATION OF FUNDAMENTAL STUDIES OF UNSTEADY TURBULENT BOUNDARY LAYERS

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Stanford University  
SI: AMES

Measurements were made on an unsteady turbulent boundary layer with well-defined initial and boundary conditions. The test boundary layer is a standard, steady, flat-plate turbulent boundary layer at the entrance to the unsteady region, and it is subjected to a linearly decreasing free-stream velocity in a time-dependent manner. The experiments were conducted in a specially designed water tunnel with the frequency of the imposed oscillations ranging from quasi-steady ( $f \rightarrow 0$ ) up to the bursting frequency in turbulent boundary layers. It was found that the mean velocity and the

turbulence intensity profiles were unaffected by the imposed oscillations. The amplitude ratio of the periodic component, although as much as 1.7 for quasi-steady oscillations, becomes unity over the outer region of the boundary layer at higher frequencies. Both the boundary layer thickness and the Reynolds stress distribution across the boundary layer become frozen over the oscillation cycle at high frequencies.

#### 16841 THREE-DIMENSIONAL TURBULENT BOUNDARY LAYER ON A BODY OF REVOLUTION AT INCIDENCE

V. C. Patel  
B. R. Ramaprian  
University of Iowa  
SL: BRL

The research objective is to conduct an experimental and theoretical study of the three-dimensional turbulent boundary layer on bodies of revolution at incidence. The experimental phase will include detailed measurements of the Reynolds stresses, direction of the wall shear stress, and the mean flow in the neighborhood of a free-vortex type of separation. The theoretical study will consider large and reversing crossflows and free-vortex type of separation.

#### 16972 PROPERTIES OF ACTUAL AND NUMERICAL SHOCK AND BLAST WAVE PHENOMENA

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SL: BRL SC: LCWSL

The objective of the research is to measure physical quantities in flows produced by two-dimensional shock waves and spherical blast waves and to develop numerical methods that will accurately predict and describe such flows. Using a hypervelocity shock tube and interferometer augmented by pressure gauges and heat-transfer gauges, oblique shock diffraction in air and spherical blast waves will be studied. The random choice method of Glimm, Chorin and Sod will be

developed for two-dimensional and spherical blast waves interacting with surfaces. In order to complement the research on monatomic and diatomic gases, a study on oblique shock wave diffractions in carbon dioxide has been completed. The latter is a gas which is already considerably vibrationally excited at room temperature and real-gas effects can be observed quite readily. Numerical work is going ahead on the domains and boundaries of oblique-shock-wave diffraction in air up to very high Mach numbers and these will be tested experimentally including nonequilibrium effects. Some experimental data has already been obtained. The Random-Choice Method has been applied to the solution of the shock-wave transition problem of a spherical blast wave, to a dusty-gas shock tube, and to oblique shock wave reflections.

#### Reports:

1. Pseudo-Stationary Oblique Shock-Wave Reflection in Carbon Dioxide - Domains and Boundaries, by Samon Ando, Apr 81, MS Thesis.
2. Weak Spherical Shock-Wave Transitions of N-Waves in Air with Vibrational Excitation, by H. Honma et al.
3. Beyond Three Decades of Continuous Research at Utias on Shock Tubes and Waves, by I. I. Glass
4. Temperature Measurements of an Implosion Focus, by T. Saito et al.
5. Response of a Room Subjected to Simulated Sonic Booms, by N. N. Wahba et al.

#### 17048 DEVELOP A NAVIER-STOKES ANALYSIS TO INVESTIGATE THE MECHANISM OF TRANSONIC SHOCK-WAVE BOUNDARY LAYER INTERACTIONS

Henry McDonald  
Scientific Research Associates, Inc.  
SL: BRL SC: AMES

The research objective is to develop a Navier-Stokes calculation procedure to investigate details of shock wave-boundary layer interaction phenomena in axisymmetric and unsteady environments. A previously developed Navier-Stokes numerical solver will be extended to include adaptive mesh capability which would continuously resolve those regions of the interaction flow field having the largest flow gradients. In addition, the program will investigate artificial shock damping formulations and turbulence model formulations.

**17134 CAVITY FLOW OSCILLATION**

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SL: HDL SC: LCWSL, BRL

The objective of the research is to determine the feedback mechanism responsible for the self-sustained oscillation in flows over shallow axisymmetric cavities ranging from subsonic to supersonic Mach numbers. An experimental study using axisymmetric cavities of various heights and depth in both an open jet facility and wind tunnel will be carried out by making cross correlations of the simultaneous measured velocity perturbations at various axial locations along the cavity shear layer and the pressure oscillations inside the cavity. The experimental research on axisymmetric cavity flow oscillations with an ellipsoidal nose is in progress. The boundary layer measurements at the upstream cavity corner were made using a laser Doppler Velocimeter (LDV). These measurements were made in a water channel using the 4" diameter axisymmetric model. These results were in agreement with the computed results obtained by Thwaites method. In order to understand the role of the initial flow conditions and the interaction of shear layer with the downstream cavity corner, two heating strips were installed to control the initial flow conditions. By applying a sinusoidal current to the heating strip, Tollmien-Schlichting (T-S) waves were excited. Because of the natural amplification of the T-S waves by the laminar boundary layer, a periodically perturbed shear layer with frequency was obtained. This range of frequencies will cover the entire first mode cavity flow oscillations.

**17226 MECHANICS OF THE REMOVAL OF THICKENED CHEMICAL AGENTS FROM CONTAMINATED SURFACES BY WIPING**

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SL: CSL SC: CSL, DPG

The theoretical and experimental base which is required to predict the behavior of polymeric fluids

on wiping flows is not adequate. The objective is to provide an experimental base and to then make predictions through theoretical analysis. The wiper will be stationary but at various angles to a flat surface. Rotating wipers will have the center of rotation stationary. A polyester film will move off a reel at a given velocity and will pass below a reservoir of the viscoelastic fluid. The polyester film transports the fluid at a constant thickness to the wiper. In this way, a stationary steady flow field will appear to the observer. (Instabilities will also be recognized depending upon fluid velocity, fluid properties, load on wiper, wiper stiffness and surface roughness). Surface roughness will be controlled by embossing the polymeric film after heating to obtain the proper degree of softening. This allows quantitatively controlled surface topology. The critical experimental measurement and theoretical prediction is the remaining film thickness. Wiper temperature will also be considered as a variable. Accomplishments to date include the following: (1) The rheometrics mechanical spectrometer has been installed in the laboratory, and has undergone extensive testing and calibration. (2) Some preliminary data have been obtained on various rheological properties of the type of fluid which will be studied. While these data appear reasonable, they are incomplete.

**17348 SUBSONIC STEADY AND UNSTEADY AERODYNAMIC LOADS ON MISSILES**

A. H. Nayfeh  
Dean T. Mook  
Virginia Polytechnic Institute and State University  
SL: MICOM

The objective of the research is to develop a continuous-vorticity technique to treat lifting and nonlifting subsonic flows for low aspect ratio wings at angles of attack and ultimately extend the study to unsteady lifting flows. Linearly varying vorticity distributed over plane triangular areas will be used to treat lifting surfaces with edge separation and the developing shed wake. Next the approach will consider finite thickness and nonlifting bodies using a surface distribution of vorticity.

### 17425 TRANSONIC BLADE-VORTEX INTERACTION NOISE AND UNSTEADY WAKE FLOW

A. R. George  
Cornell University  
SL: AMES

The objective of the research is to develop simplified analysis techniques for understanding the principal features of noise radiation from the interaction of a transonic rotor tip with a vortex shed from a previous blade passage. Perturbations of wake geometry due to turbulence efforts will be carried out to predict changes in vortex geometry and the transonic flow will be analyzed by a high reduced frequency linearization about the steady transonic flow. Resulting outgoing sound waves will be modeled by either classical Lighthill techniques, geometrical acoustics or Kirchhoff type formulation. Efforts have primarily been given to studying the applicability of various transonic flow analyses to a helicopter advancing blade interacting with a nearly parallel tip vortex. An intensive literature survey was made of both experimental data and of existing unsteady transonic flow computation methods. The various data support the transonic character of the interaction and it is clearly very important whether the vortex "induced velocity" adds to or subtracts from the axial free stream flow velocity of the blade. This has been identified as important in both single rotor in tandem blade interactions.

### 17690 CONCEPTUAL EXPERIMENTS IN TRANSITION OF TURBULENCE THROUGH COMBINED VISUALIZATION AND HOT-WIRE TECHNIQUES

M. V Morkovin  
Illinois Institute of Technology  
SL: BRL SC: HDL, AMES

Basic conceptual issues in stability and transition in shear layers which relate to roughness effects, wave amplification in boundary layers and transverse turbulent contamination will be examined through combined utilization of advanced visualization techniques and hot-wire anemometry. Movable smoke sheets will be used to furnish global

information about the nature of non-linear regions of disturbances and traversing hot-wire probes, guided by the visual information, will map out the local character and growth of shear-layer disturbances and breakdown regions.

### 17698 COMPUTER TOMOGRAPHY FOR AERODYNAMIC MEASUREMENTS

Charles M. Vest  
University of Michigan  
SL: LANGLEY, AMES

The objective of the research is to develop multidirectional holographic interferometry with data analysis by computer tomography for application to complex aerodynamic flows. Instantaneously recorded interferometric data will be photometrically read out, digitized and then processed by a computer to yield density maps on a set of planar cross sections of a three-dimensional flow. Development of computer tomography codes, holographic interferogram scanner and data preprocessor, and the execution of two prototype experiments are included.

#### Reports:

1. Tomographic Reconstruction of Strongly Refracting Fields and Its Application to Interferometric Measurement of Boundary Layers, by Soyoungh Cha et al, *Appl Opt.*

### 17747 NUMERICAL MODEL FOR FLUID BEHAVIOR DURING SPIN-UP OF LIQUID-FILLED SHELLS

G. F. Homicz  
Calspan Corporation  
SL: BRL, CSL, LCWSL

The research objective is to develop a partially phenomenological model and a numerical solution for the fluid mechanical processes occurring in the spin-up of liquid-filled shells. In the first phase, a phenomenological model will be developed for predicting the unsteady flow during spin-up of the liquid in a partially filled cylinder, including situations where the free surface intersects one or both of the endwalls, while in the second phase the spin-up and stability problems would be attacked



starting from first principles, i.e., a finite difference method for solving the unsteady, three-dimensional Navier-Stokes equations.

## 17778 SECOND PHASE OF MUZZLE BLAST FLOW

Gino Moretti  
G.M.A.F., Inc.  
SL: BRL SC: LCWSL, BWL

The research objective is to numerically solve for the fluid dynamic processes occurring during the exit of a shell from a barrel properly accounting for the region occupied by relatively cold air being perturbed by the forward motion of the projectile and the region occupied by the burned gases at high temperature and pressures exhausting after the projectile has emerged from the muzzle. Two separate but similar computational programs reflecting the proper thermodynamics and boundary conditions for the two regions will be written for solving the governing Navier-Stokes equation utilizing a conformal mapping technique and integration schemes developed in a previous study on the precursor flow.

## 18000 COMPUTATION OF VISCOUS SUPERSONIC FLOW FIELDS ABOUT ADVANCED PROJECTILES

J. L. Steger  
Stanford University  
SL: BRL SC: LCWSL

The research objective is to develop numerical techniques for accurately and efficiently solving the problem of supersonic steady and unsteady flow past axisymmetric and nonaxisymmetric bodies. A parabolized Navier-Stokes code will be modified to conduct studies of the subsonic sublayer approximation. An efficient grid generation method will be developed for nonaxisymmetric cross sections and changes to the numerical algorithm will be formulated for finned projectiles. In addition, a new unsteady algorithm will be developed for unsteady supersonic flows.

## C. COMBUSTION

### 13653 BASIC EROSION INVESTIGATION IN SMALL TURBOMACHINERY

Widen Tabakoff  
University of Cincinnati  
SL: EUSTIS

The particle rebound measurements and analysis were completed. Initially rebound data has been obtained for 2024 Al, Ti 6-4, and INCO 718 sample materials at various particle velocities, impingement angles and temperatures. Virtually no differences in rebound characteristics were found among the three tested materials. To further verify this result, particle rebound characteristics were measured for lead as a target material. Lead was used as it substantially expanded the range of the yield strength of the materials tested. As stated above no significant change was measured among the target materials tested including lead. The particle rebound results are presently being evaluated for their impact on the erosion rate model. In this respect, the rebound restitution ratios have been converted into work input into the target material. Although the restitution ratios were not found to vary with material, they did vary radically with impingement angle as does the erosion rate.

#### Reports:

- No. 1-3 in previous editions.
- 4. Temperature Effects on the Erosion of Metals Used in Turbomachinery, by W. Tabakoff et al, *pres 5th Intl Conf on Erosion by Liquid and Solid Impact*.
- 5. Particle Dynamics and Erosion in Turbomachinery, by W. Tabakoff, *pres at 79 Intl Joint Gas Turbine Congress and Exhibition*.
- 6. Test Facility for Material Erosion at High Temperature, by W. Tabakoff et al, *Erosion: Prevention and Useful Appl* p123 (1979). AD A075 030
- 7. Erosion Behavior in a Simulated Jet Engine Environment, by T. Wakeman et al, *J Aircraft* 16, 828 (1978).
- 8. Effects of Temperature on the Behavior of Metals Under Erosion by Particulate Matter, by Nahum Gat et al, *J Testing and Evaluation*, 177 (1980). AD A088 868

### 13935 HIGH PRESSURE ATOMIZATION

F. V. Bracco  
Princeton University  
SL: AFRL SC: TASDC

The main objective has been to study the fundamental processes which lead to the

atomization of high pressure liquid jets injected into compressed gases through single hole nozzles. Work to date has resulted in identification of the mechanism even though some additional testing is advisable.

#### 15048 WALL EFFECTS ON COMBUSTION IN AN ENGINE

Francis E. Fendell  
TRW, Inc.

SL: AFLRL, AMMRC SC: TARADCOM

With respect to activities carried out previously, first some inferential evidence concerning the thickness of the oil film left on a engine cylinder side wall owing to reciprocating-piston motion has been obtained. With the reduction of heat transfer from the proposed ceramic engines and the accompanying higher wall temperatures (perhaps six-tenths of typical adiabatic flame temperatures for hydrocarbon-air reactants), the required properties of the high-molecular weight oils may be influenced by the residual oil sheath. It may be shown that over a wide range of rpm an oil layer of only one-to-two microns might well become saturated with unburned hydrocarbon gas under typical operating conditions, whereas a ten-micron thick oil layer probably would not. In other words, the diffusional resistance of the liquid to gaseous absorption (as described at the oil-film/bulk-gas interface by Henry's Law) is significant for a thicker layer. Further it may be shown that if (and only if) diffusional resistance is significant is the level of absorption/desorption from the oil film (and presumably unburned-hydrocarbon-exhaust levels from the cylinder as well, since the desorption of unburned fuel vapor from the oil film may occur so late in the engine cycle that oxidation with expansionally cooled bulk gases is precluded) sensitive to rpm. Now, in fact, the unburned-hydrocarbon-exhaust levels from conventional engines are exceptionally sensitive to rpm; the modest amount of wall temperatures possible at large horsepower output does not explain the decrease in HC levels with increase in rpm. Thus, tentatively the inference is drawn that oil films on cylinder walls may be closer to ten microns in thickness than one micron.

#### Reports:

No 1-7 in previous editions.

8. Cyclic Absorption/Desorption of Gas in a Liquid Wall Film, by George Carrier et al, *Combust Sci Tech* 25, 9 (1981). AD A099 653

#### 15403 STUDY OF HYDROCARBON FORMATION IN THE TEXACO COMBUSTION SYSTEM BY IN-CYLINDER SAMPLING

P. S. Myers  
O. A. Uyehara  
University of Wisconsin at Madison  
SL: TARADCOM SC: AFLRL

A variety of sampling experiments have been performed. Because of past difficulties associated with the reciprocating motion of the sampling valve, it was decided to first sample with the valve fixed at a number of positions within the clearance volume. Successful completion of this series of tests was followed by reconnection of the valve drive linkage with the penetration ratio (the ratio of sampling valve to piston motion) set at its smallest value, (i.e. 1/3) and initiation of a similar series of sampling tests which are currently underway. The penetration ratio will be gradually increased and new tests run until has been reached correspondence between the valve and piston motions. In this way it is hoped to maximize the amount of information obtained prior to any future mechanical failure. Sample handling procedures have been modified in response to two analysis problems which have occurred. A portion of the sample from the valve is now being run through an oxidizing catalyst (CuO) to complete combustion. Analysis of this stream results in an expression for overall air/fuel ratio which is dependent only on the CO<sub>2</sub> measurement and yields more consistent results.

#### 15532 ACOUSTIC SIGNATURE FROM FLAMES AS A COMBUSTION DIAGNOSTIC TOOL

Warren C. Strahle  
Georgia Institute of Technology  
SL: BMDATC, LEWIS SC: TARADCOM

Conversion of the gas turbine combustor injection system for operation on propane was completed.

An initial attempt at measuring a cross section average of the ion density was accomplished by using a stainless steel wire mesh as one electrode and the combustor wall as the second electrode. Saturation current was achieved at a potential of about 100 volts and axial profiles of the mean and fluctuating saturation current were obtained. The probe is being modified for use of a Pt-Rd mesh to withstand higher fuel/air ratios. The theory for the acoustic fluctuations in the gas turbine has been worked out and the decision has been made to measure the terms in the governing differential equation, rather than to attempt an integral equation inversion. This decision has been guided by the extreme difficulty encountered in the inversion of the two dimensional integral equation for the case of the premixed flame in an anechoic chamber. In the latter case, final analytical work is in progress and it has been found that satisfactory numerical results can only be achieved for the one-dimensional case. Consequently, the information which will be achieved for the open flame will be the axial distribution of the product of the heat release rate fluctuation and the correlation length scale. It was originally hoped that each item could be separately determined from the acoustic measurements.

*Reports:*

No. 1 in previous edition.

#### 15662 A STUDY OF THE MECHANISM OF LEAN LIMIT FLAMMABILITY

Roger A. Strehlow  
University of Illinois, at Urbana  
SL: AFRL, TARADCOM

A considerable amount of data has been accumulated. Schlieren pictures of upward propagating flames have been obtained for the 50 x 50 mm, 100 x 100 mm, 200 x 20 mm, and 20 x 200 mm tubes. It has been observed that upward propagating flames are cellularly unstable in the direction of any tube dimension that is higher than 50 mm. Upward propagating velocities have also been measured for all these cases over a range of lean limit compositions up to the extinguishment point. The upward propagation speeds at the lean limit all correlate very well with the Collins equation for the upward propagation of a hot

bubble of gas in the tube containing a colder high density gas. Composition measurements have been obtained with a small sample probe which draws approximately 1 cc of gas from a location either ahead of, in, or behind the flame, but after some review, the studies of the gas composition ahead of and behind the flame has been discontinued because of the realization that the sample size is just too large when compared to the flame thickness to allow any definite conclusions concerning preferential diffusion or other processes occurring in the flame.

#### 15788 DIESEL COMBUSTION ANALYSIS USING RAPID SAMPLING TECHNIQUES

G. L. Borman  
O. A. Uyehara  
P. S. Myers  
University of Wisconsin, at Madison  
SL: TARADCOM SC: AMMRC

The purpose of the research is to obtain a better understanding of the fundamental processes which take place in a diesel cylinder during combustion. Two experimental methods are being used. In the first method a small sample of gas is removed from the cylinder by a sampling probe. The second method, called dumping, cuts a diaphragm in the cylinder head at a given time so that the cylinder gas rapidly expands into a collection chamber. Repeating the dumping at various times allows a history of the formation rate of a given species to be obtained. Work was completed on taking additional sampling and dumping data. In particular, dumping data was taken with a four hole nozzle at engine conditions of load and speed the same as those previously gathered with the eight hole nozzle. During this data gathering it was noted that engine vibration was taking place at higher loads. Engine main bearings were found to have excessive clearance and have to be replaced. The electronic fuel injection system was received and is being set up.

*Reports:*

1. Time- and Space-Resolved Species Determination in Diesel Combustion Using Continuous Flow Gas Sampling, by K. E. Rhee et al. *Soc. Automotive Eng. Inc.* p 959 (1979) #780226.

### 16791 SHOCK-TUBE STUDIES OF IMPORTANT REACTION STEPS INVOLVING $\text{CF}_3\text{Br}$ FOR THE INHIBITION OF HYDROCARBON COMBUSTION PROCESSES IN AIR

K. G. P. Sulzmann

University of California, at San Diego  
SC: BRL, AFLRL, MERADCOM

A He-Xe laser and a liquid-nitrogen cooled InSb infrared detector have been installed for near line-center absorption measurements on shock-heated  $\text{CH}_2\text{O}$  near  $3.508\mu$  by a triple path method. In addition, two existing infrared monochromators have been fitted with NaCl-prisms and with liquid nitrogen cooled infrared detectors (InSb and HgCdTe) for absolute infrared emission measurements involving simultaneously two of the following alternate species:  $\text{NO}_2$  near  $7.58\mu$  and/or near  $6.25\mu$ ,  $\text{HNO}_2$  near  $7.91\mu$  and/or  $5.89\mu$  and/or  $2.79\mu$ ,  $\text{N}_2\text{O}$  near  $7.78\mu$  and/or near  $4.52\mu$ ,  $\text{NO}$  near  $5.33\mu$ ,  $\text{CO}$  near  $4.80\mu$ ,  $\text{CO}_2$  near  $4.25\mu$ ,  $\text{H}_2\text{O}$  near  $6.27\mu$  and/or near  $2.66\mu$ , and  $\text{H}_2\text{CO}$  near  $3.51\mu$ . An available system for the production of formaldehyde monomer from paraformaldehyde has been re-activated and is presently being tested. All test gases and chemicals are available for  $\text{CH}_2\text{O}-\text{NO}_2-\text{Ar}$  runs.

#### Reports:

1. Approximate Analytical Relations for Radiative Recombination of Br-Atoms by Free-Bound Transitions, by K. G. P. Sulzmann et al, *J Quant Spectrosc Radiat Transfer*.

### 16914 SURFACE IGNITION AND HETEROGENEOUS CATALYSIS OF HYDROCARBON FUELS

Richard I. Masel

R. O. Buckius

University of Illinois, at Urbana  
SL: TARADCOM

This research project consists of a parallel experimental and analytical study to investigate catalytic combustion for piston engine applications. The broad objectives are to determine reaction rate data for various catalysts, to investigate the heterogeneous reactive fluid mechanics in flow systems, and to analytically model such phenomena. The ignition experiments

performed to date have used platinum and oxidized nickel as catalysts and propane, propylene, methanol, ethanol, and toluene as fuels. The results of propylene, methanol, ethanol, and toluene indicate that the catalysts reduce the ignition temperatures by as much as 100 degrees C. A rather unusual result which has been observed with propylene is that an apparent ignition occurs in a mixture which is too lean to burn according to standard tables. Initial kinetics experiments utilized propylene oxidation on a nickel oxide catalyst, but the kinetics of the reaction were much more complicated than originally expected. Current measurements are considering a wider variety of catalysts and fuels and the current kinetics efforts are considering some of the simpler intermediates produced. The analytical effort is directed towards a two-dimensional transient investigation. The governing equations for the time dependent, two-dimensional problem including heterogeneous reactions have been formulated including species diffusion. The highly differing rates of the reactions must be included into the numerical scheme. These initial calculations considering unsteady channel flow have achieved some success yet the computational effort is far too large when extended to the reaction calculations. Present efforts consider the computational efficiency of simplified problems.

#### Reports:

1. The Effect of Surface Protrusions on Self-Sustained Thermal Oscillations During Hydrogen Oxidation on a Nickel Foil, by A. G. Sault et al, *J Catalysis*.

### 16957 PREIGNITION OXIDATION CHARACTERISTICS OF HYDROCARBON FUELS

R. S. Cohen

N. P. Cernansky

Drexel University

SL: MERADCOM SC: TARADCOM

An experimental program to study the preignition oxidation characteristics of hydrocarbon fuels has been undertaken. The basic approach for the program is to use chromatographic profiling and analysis techniques to qualitatively and quantitatively assess the reactants, products, and stable reaction intermediates during the preignition



oxidation process. These species are being followed in time up to the point of hot ignition in a static reactor test facility, which is being exercised over a range of operating conditions. Analysis of these data should lead to an understanding of the global preignition oxidation mechanisms and associated heat release rates, along with identification of the important fuel and physical factors determining the preignition behavior. One task centered around facilities and equipment improvements, including: a general refurbishing of the static reactor facility, and setting up and debugging the chromatographic and peak profiling data systems. The other task involved preliminary testing for system verification.

#### 16983 IGNITION AND UNSTEADY FLAME PROPAGATION IN TURBULENT REACTIVE FLOWS

T. Y. Toong  
Massachusetts Institute of Technology  
SC: TARADCOM, MERADCOM

The research objective is to experimentally examine ignition and unsteady flame propagation in turbulent reactive flows and to compare the results with the theoretical prediction available in the literature. New theoretical studies will be undertaken to explain any discrepancies noted in these comparisons. Two research phases are proposed, the first will be on the flame-turbulence interaction, the second on the ignition in turbulent flow. Analysis and theoretical modeling will be secured through experiments conducted with turbulent flat flames using hot-wire anemometry and Schlieren photography. The mean gas temperature will be measured by means of fine thermocoupler and resistance thermometers. These data will then be compared with experimental data of other investigators who have reported their findings using other techniques in the field. Major effort has been devoted to (1) the design and the construction of a flat-flame burner and (2) a critical assessment of the effects of the turbulence scales and intensities on flame propagation. The burner consists of a vortex mixing chamber, a settling chamber, a flow conditioning section and a combustion chamber. Various correlations have been examined among the measured apparent turbulent flame propagation speeds, the laminar speeds and the Reynolds numbers based on the

turbulence intensities, micro-scales or integral scales.

#### 17001 IGNITION AND FLAME PROPAGATION IN SPRAYS

W. A. Sirignano  
S. C. Yao  
Carnegie-Mellon University  
SL: AMES, LEWIS SC: TARADCOM, MERADCOM, AMES

The research objective is to investigate experimentally the combustion of hydrocarbon droplet sprays in air. It is planned to measure the effect of droplet diameter, droplet number density, overall equivalence ratio and fuel type on the flame speed, flammability limits, and flame propagation mechanism. By means of additions to the model (a numerical computer of laminar spray combustion), the model predictions will be compared with experimental data currently available in the literature and those measurements obtained from constructing the experimental apparatus. This then introduces a new method of forming a one-dimensional spray. The details and results of one-dimensional heterogeneous turbulent flame propagation model in an enclosed chamber were presented at the ASME 1980 Winter Annual Meeting in Chicago. Currently, the efforts are being continued to study the phenomena of heterogeneous flame stabilization. The purpose is to obtain a stationary flame for a mixture of air-fuel drop-fuel-spray flowing in a one-dimensional open chamber. The impulse-jet spray generator has been fabricated. The droplets are ejected in a frequency range 10-1000 Hz. The droplet size is on the order of 400  $\mu\text{m}$ . The second generation of impulse-jet generators is under design. The piezoelectric capability should be greatly increased to overcome the surface tension in ejecting droplets of 100  $\mu\text{m}$ .

#### 17034 ANALYSIS OF THE INTERIOR BALLISTICS PROCESSES OF BULK LOADED LIQUID PROPELLANT GUNS (LPG)

Raymond B. Edelman  
Science Applications, Inc.  
SL: BRL SC: ICWSL

The purpose of this investigation is to develop a versatile engineering model of the liquid propellant

guns (LPG) combustion process. The model will provide a user oriented research tool that can establish the sensitivities of the pressure-time history to each of the mechanisms involved in the process and to provide the framework of a predictive tool that will be suggestive of design features necessary for controlled combustion and gun features for satisfactory operation. During the first year of the program a model applicable to the analysis of the interior ballistics processes of liquid propellant guns was formulated and preliminary applications of the model were made. The model formulation is based upon a system of transient, two-dimensional conservation equations for mass, momentum, energy and species. This model represents a framework within which a broad range of fundamental mechanisms are being investigated. Turbulent mixing, chemical kinetics, wave propagation and multi-phase flow phenomena are included in the study. Various state-of-the-art and advanced numerical methods are being investigated. Studies during this first year show that for the widely varying flow speeds and widely varying time scales of wave propagation, chemical reactions, convection, and turbulent mixing that explicit or semi-explicit methods are appropriate for accuracy requirements demanded by these widely varying time scales; however, other advanced methods including but not limited to linearized block implicit methods are also being studied. This analysis is structured to be applicable to a wide range of multi-phase flow problems. Preliminary applications of the present model and numerical technique have reproduced an analytical solution of the Lagrange gun problem. This comparison demonstrated the correctness of the conservation equations and the accuracy of the numerical technique for this problem.

#### 17062 BASIC STUDY OF FUEL STORAGE STABILITY

F. R. Mayo  
Theodore Mill  
S. E. Buttrill, Jr.  
SRI International  
SI-MERADCOM

The research objective is to investigate fuel instability over a period of three years. This will involve studies such as free radical oxidation of fuels, detection of deposit precursors, homogeneous and heterogeneous metal catalysis,

effect of sulfur, nitrogen and peroxide compounds, stabilization of fuels, and predicting and improving stability. Fifteen diesel fuels were received from the U. S. Army Fuels and Lubricants Research Laboratory. Their origins and their total insolubles according to accelerated stability test D 2274 were also supplied. Five of these fuels, with total insolubles ranging from 0.4 to 10.0 mg/100 mL were distilled at about 2.1 kPa (16 torr) and stored under nitrogen so that they can be compared under unaged conditions (which may not correspond with the total insolubles). Trials of oxidations are being made to determine the lowest temperature (nearest to storage conditions) at which a convenient rate of oxidation could be obtained.

#### 17247 INVESTIGATION OF THE IGNITION SENSITIVITY OF PROPELLANTS TO PROJECTILE IMPACT

W. Hoyt Andersen  
Shock Hydrodynamics  
SI: BRL, LCWSL

The objective of this research is to continue the current analysis so as to complete the overall quantitative description of the impact ignition event so that it will be useful for correlation and predictive purposes. This requires investigation of details of the fundamental mechanism involved, including the role of heterogeneous defects (particle size and porosity) on the ignition and reaction build up processes. Analytical and experimental studies will determine the effect of particle size and porosity on the impact ignition characteristics (such as LOVA-XI propellant) of propellants. Characteristics such as the thermal decomposition kinetics, reaction rates, hot spots in the impacted propellant, projectile diameter, increase of shock temperature due to porosity, etc., will be obtained experimentally. These attributes will be used to formulate and perfect the overall model on a numerical basis. Propellant samples for the impact tests are currently being made. Studies have been conducted to place the previously postulated impact ignition model on a more quantitative basis, and elucidate the controlling parameters. Analytical studies are in progress to determine the shock Hugoniot and the thermal energy partition in the individual ingredients of an impacted propellant that is made up of more than one

component. It was shown that the detonation velocity and pressure of explosives and propellants can be estimated to good approximation by simple equations that are based on the conventional gamma law state equation, using an empirical adiabatic exponent whose value is largely a linear function of the charge density of the explosive.

#### Reports:

1. Detonation Characteristics, Shock and Impact Sensitivity of Low Vulnerability Ammunition Propellant, by W. H. Andersen et al, *Proc 5th Symp on Vulnerability and Survivability of Aerial and Surface Target*.
2. Surface Ignition of Explosives and Propellants by a Hot, Stagnant Gas Pocket, by W. H. Andersen et al, *Combust Sci Tech* 24, 35 (1980). AD A095 632
3. Model of Impact Ignition and Explanation of Critical Shock Initiation Energy. II. Application by W. H. Andersen, *Proc 7th Symp on Detonation*.
4. Comments on "The Chemistry of Detonations", by W. H. Andersen.

### 17267 AN INVESTIGATION OF THE KINETICS OF CATALYZED COMBUSTION OF FUEL

Pierre J. Marteney  
United Technologies Research Center  
SL: LEWIS

The research objective is to determine the feasibility and efficiency of using catalyzed surface reactors in the combustion of various fuels through a fundamental understanding of the reaction rates and mechanisms on the surface. An experimental and analytical program will examine the effects of catalyst type and loading, substrate geometry, temperature, stoichiometry, residence time, and heat loss or gain on reaction rates for propane and other fuels. Samples for this program have been prepared and one of each type is mounted and instrumented with thermocouples. With this combination of samples (8), it is possible to determine the effects of surface-to-volume ratio, concentration of active metal, and cell diameter and shape on the catalytic reaction. Experimental measurements include the inlet and outlet concentrations, inlet and outlet temperatures, and the bed temperature profile.

These data, in conjunction with the analytical model of the catalytic combustor, yield the stepwise rates of conversion of propane to carbon monoxide to carbon dioxide on the surface. The rate of reaction of carbon monoxide is determined in separate tests. Gas phase rates for propane and CO oxidation are taken from the literature.

#### Reports:

1. Kinetics of Surface Reactions in Catalytic Combustion, by P. J. Marteney et al, *pres 18th Combust Symp.*

### 17378 STRUCTURE AND CHARACTERISTICS OF HETEROGENEOUS DETONATION

J. A. Nicholls  
Martin Sichel  
University of Michigan  
SL: LCWSL SC: BRI

The research objective is to elucidate the controlling mechanisms in the initiation and propagation characteristics of heterogeneous detonation. The analytic and experimental study will examine the following aspects of heterogeneous detonation: extended droplet sizes and equivalence ratios, velocity deficit, vapor pressure, wave structure, cloud gaps, and critical initiation energy. A finite difference code, originally developed by Eidelman and Burcat (AIAA J 18, 1103, 1980) has been used to compute the static and dynamic impulse generated by detonations propagating through decane oxygen mixtures. The results were compared to the impulse generated in wheat air and RDX air mixtures in order to establish the influence of particle loading and heat of combustion upon impulse. The same code was also used for a detailed investigation of the structure of two phase detonations during the transition from an initiating blast wave to a fully established detonation.

#### Reports:

1. The Transitional Structure of Detonation Waves in Multi-Phase Reactive Media, by Shmuel Eidelman et al, *Combust Sci Tech*.

**17459 MONTE CARLO CALCULATIONS  
OF TWO-DIMENSIONAL  
COMBUSTOR FLOWS**

Stephen B. Pope  
Massachusetts Institute of Technology  
SL: LEWIS

The research objective is to develop and demonstrate a Monte Carlo method for calculating two-dimensional recirculating turbulent combustion flow fields. A combined Monte Carlo/finite-difference procedure will be used to calculate the velocity field and specie concentrations in two-dimensional combustor configurations. The models will include finite-rate hydrocarbon and NO<sub>x</sub> chemistry, and the calculations will be compared with existing data.

**18188 DIESEL ENGINE CYLINDER GAS-  
SIDE HEAT FLUX TO A CERAMIC  
SURFACE**

Gary L. Borman  
University of Wisconsin  
SL: TACOM SC: MERADCOM

The research objective is to obtain an improved understanding of the heat transfer phenomena in adiabatic diesel engines and to provide improved correlations for use in cycle simulation and combustion modeling of such engines. A single cylinder TACOM-LABECO research engine with a special head providing

a one inch diameter cylindrical port access to the combustion chamber will be fitted with a ceramic disk in this access port such that thin film platinum resistance transducers can be mounted on the gas side and on the coolant side of the ceramic disk. Disk surface temperatures will be obtained under both motoring and firing conditions and techniques established to separate convective and radiative components of the total heat flux.

**18333 HIGH PRESSURE ATOMIZATION  
AND THICK SPRAYS**

F. V. Bracco  
Princeton University  
SC: TACOM. MERADCOM. AFRL

The research objective is to test the newly developed and unique model for thick sprays and to confirm the validity of the supplemented aerodynamic-interaction mechanism of liquid jet breakup in the atomization regime. Experiments utilizing a laser velocimeter and optical techniques together with theoretical analysis will be performed to further determine the effects of ultra high liquid injection pressures (up to 30,000 psi); straight tube sharp edge inlet nozzles with length to diameter ratios in the range of 2-10, and heated cylinder gas (up to 750 degrees K) on liquid jet atomization and subsequent motion and dynamics of thick sprays.



## IV METALLURGY AND MATERIALS SCIENCE

### A. DEGRADATION AND REACTIVITY

#### 14679 BONDING AND INTERACTION OF ATOMS AND MOLECULES ON SINGLE CRYSTAL SURFACES

William R. Graham  
Max E. Caspari  
J. Robert Schrieffer  
University of Pennsylvania  
SC: MERADCOM, BWL, AMMRC

Studies of hafnium (Hf) adsorption on the (111), (211) and (321) surfaces of tungsten have been continued using field ion microscopy. *W(111)*: Individual hafnium atom adsorption site geometry has been found to be accessible through direct imaging. From a series of 115 fairly well resolved individual adsorption events with Hf, it can be concluded that in each case the adsorption site geometry corresponded to a normal, equilibrium, lattice site position. In the case of Hf adsorption on larger *W(111)* planes color superposition techniques have been used to confirm this conclusion. *W(211)* and *W(321)*: Sets of 30-40 diffusion intervals at each temperature have been obtained. Utilizing the color superposition technique these observations provide the mean square displacement as a function of temperature. Using the Arrhenius equation, diffusion coefficients were obtained. Other research has centered upon the task of working out the theoretical expression for a gamma-gamma perturbed angular correlation, for the special case in which one of the gamma rays travels along the direction normal to the crystalline x-y plane. This theoretical expression is now in the process of being transformed into a useful numerical form with the aid of a computer.

#### 14740 IMPROVED MOISTURE RESISTANCE OF FIBER REINFORCED PLASTICS

Jack L. Koenig  
Case Western Reserve University  
SL: AMMRC, NLABS SC: LANGLEY, EUSTIS,  
MERADCOM, MICOM

Previous work indicated that the number of active amino groups of the coupling agent determines the

amount of interfacial bonding at the interface of matrix and coupling agent. In order to improve the adhesion between epoxy matrix and glass fiber, two multiple aminosilanes have been investigated, one is N-2-aminoethyl-3-aminopropyltrimethoxysilane and the other is trimethoxysilylpropyldiethylenetriamine. Fourier transform infrared spectroscopy was used to determine the molecular structure of these multiple aminosilane coupling agents adsorbed onto glass surfaces. Results indicated that the adsorption phenomena of the aminosilanes onto glass surfaces from aqueous and organic solvents are quite different. In fiber-reinforced composites the interfacial bonding between fiber and matrix can influence various aspects of composite behavior. In order to understand the role of matrix interfacial bonding on composite performance, a fiber pull-out testing method is being used. This method, in addition to its relative simplicity of sample preparation and measurement, is expected to give realistic information of the debonding at the interfaces in fiber composites.

#### Reports:

- No. 1-2 in previous editions.  
3. Fourier Transform Infrared Spectroscopic Study of the Adsorption of Multiple Amino Silane Coupling Agents on Glass Surface, by Chwan-hwa Chiang et al, *J Colloid and Interface Sci.*

#### 14766 CALCULATIONS OF SURFACE BINDING AND MOBILITY OF ADATOMS

H. B. Huntington  
Rensselaer Polytechnic Institute  
SL: AMMRC, BWL

Work on writing a paper on "Surface Ledge for a Jellium Metal" is in progress. The main result of the calculation has been to show that the adatom on the surface acquires additional binding on approaching the ledge from the lower terrace but becomes less well bound on approaching the ledge from the upper terrace (balustrade effect). This change in binding energy is in each case approximately 40

percent of the adatom binding to the flat surface for the square-cut ledge. It is somewhat reduced for ledges with less abrupt faces. The picture that evolves from this result is that stepped surfaces, when growing by deposition from the vapor phase, will tend to go towards the condition where the stepped planes are of equal width. In the process of the determination of the equilibrium electron charge distribution about the ledges, preparatory to the consideration of adatom binding, the influence of the ledge frequency on the surface dipole layer and the surface energy for the jellium metal, have been explored topics which the paper will also touch on.

*Reports:*

No. 1-2 in previous edition.

#### 14925 SULFUR IN THE CORROSION OF SUPERALLOYS

J. B. Wagner  
Arizona State University  
SC: LEWIS, BRL, DOE

Research has continued on the diffusion of S35 in calcia stabilized zirconia (CSZ). Diffusion studies on Te-127 were untimely discontinued because vendors would not supply this isotope any longer. Theoretical calculations on the electrical conductivity of  $Y_2O_3-ZrO_2$  have been initiated. Research on the  $SO_2-O_2$  corrosion of nickel has continued. Polycrystalline discs of CSZ of varying CaO content (9, 13, 15, and 18 mole%) have been used as diffusion sinks in the temperature range of 800-1200 degrees C. Attempts were made to test that the penetration profiles exhibited the expected  $t^{1/2}$  dependence. This behavior was not found. Theoretical calculations of electrical conductivity of  $Y_2O_3-ZrO_2$  have been begun. The general trends are similar to those published for  $CaO-ZrO_2$ . Work continues on this problem and  $Y_2O_3-ZrO_2$  pellets have been obtained for future diffusion studies. The  $SO_2-O_2$  corrosion studies continue.

*Reports:*

No. 1 in previous edition.

#### 15489 ENHANCED CORROSION RESISTANCE OF METAL SURFACES BY ION TREATMENT

Ray B. Benson, Jr.  
North Carolina State University

The entire conditions of corrosion passivation, and immunity to corrosion as a function of

electrochemical potential and pH have been established for specimens of iron, both implanted with chromium and non-implanted, in both distilled water (nil chloride) and 0.1 M NaCl solutions. The following results have been obtained: (1) For the nil chloride exposure, the potential up to which iron is immune to corrosion was improved by 100mV by implantation of chromium (150 keV,  $10^{17}$  ions/cm<sup>2</sup>) throughout the range of pH tested (2-12). (2) For the nil chloride exposure, Cr implantation resulted in extension of the passivation of iron to lower pH (stable at pH ~ 4 due to the stability of  $Cr_2O_3$  in this region). (3) For the 0.1 M chloride exposure, a similar improvement in the limiting potential of immunity of 100 mV has been observed. The extent of the range of passivation is not changed, due to the vulnerability of the  $Cr_2O_3$  oxide film to attack by Cl<sup>-</sup>. In-depth concentration profiling of the implanted Cr is being carried out by Auger-sputtering analysis in order to determine the concentration of Cr at the surface. A transmission electron microscopy study of damage to microstructure by ion implantation and its effect on the corrosion behavior of iron has been initiated. Zone-refined pure iron specimens have been rolled, annealed, and chemically polished.

*Reports:*

1. The Influence of Ion Implanted Chromium on the Electrochemical Behavior of Iron in Aqueous Solutions, by P. A. Parrish et al, *Corrosion*.
2. Enhanced Corrosion Resistance of Metal Surfaces by Ion Implantation, by Sangwook Kim, 1980, figures, MS Thesis.

#### 15539 LOCALIZED ELECTROCHEMICAL CORROSION CELLS

B. F. Brown  
R. T. Foley  
American University  
SL: MERADCOM, AMMRC SC: ARRADCOM, MICOM

Infrared absorption studies are nearly complete; however, the absorption band at 2400 cm<sup>-1</sup> is being studied more extensively to test the assignment of an Al-O-Al unsymmetrical stretch. The absorption band found near 3500 cm<sup>-1</sup>, probably assignable to polymeric species, is probably a function of pH, but this remains to be established. The ultraviolet

studies reveal a characteristic absorption at 240 nm which is currently attributed to  $\text{Al}(\text{OH})(\text{H}_2\text{O})_5^+$  and not  $\text{Al}(\text{H}_2\text{O})_6^{3+}$  as some have thought. Evidence indicates that chloride and perchlorate do not enter into the first coordination sphere of  $\text{Al}^{3+}$ . In addition, it seems likely that sulfate and nitrate do exchange with water to form complexes such as  $\text{Al}(\text{SO}_4)(\text{H}_2\text{O})_5^+$ . The freeze method of studying the local corrodent in stress corrosion cracking appears to promise more information than has been extracted with it to date. At open circuit potential, aluminum alloy 7075 in 1M NaCl appears to have roughly 0.3M Al inside the stress corrosion cracks, as contrasted with roughly saturated  $\text{FeCl}_2$  inside cracks in high strength steels. The effects of potential on SCC in aluminum in chloride environments are complicated, but it appears that at a potential of -1.3 v (SCE) the cracking rate is drastically reduced, but "cathodic" corrosion of aluminum is still observed at that potential. At such potentials soluble aluminum ions appear to exist at very much reduced concentrations, as deduced from approximate analysis of frozen corrodent.

#### Reports:

No. 1-2 in previous edition.

3. Infrared Spectra of Aluminum Salt Solutions, by J. F. McIntyre et al, Jan 81, 27p, tables, TR. AD A096 766

### 15812 AN INVESTIGATION OF THE MECHANISMS OF HOT, MULTI-PHASE EROSION/CORROSION PHENOMENA

John W. Kury  
Lawrence Livermore Laboratory  
SI: BRL, BWL SC: MICOM

Current investigations include the degradation of high melting temperature solid material surfaces when subjected to repeated pulsed heating and stress in a variety of reactive gas environments, detailed analysis of the transient solid surface states and phases which develop when exposed to hot, reacting, viscous-turbulent shear flow, and theoretical-experimental analysis of the potential role that solid particle suspensions play in either augmenting or reducing erosive heat, mass, and momentum transport in combustion flows. In the first two topics of investigation, the goal is to characterize detailed material response to a variety of erosive combustion flow conditions. Efforts are

being made to define erosive-resistant elemental and composite solid materials and coatings as well as special preparation and fabrication processes which will enhance the material's ability to resist erosion/corrosion. In the last topic, the influence of additives on suppressing surface erosion is being examined with respect to mechanisms at the solid surface as well as within the driving combustion process, itself. The goal is to define particle influences on combustion-induced erosive transport with particular attention to the effects of particle size distribution, placement and population distribution in the flow, chemical composition and thermo-chemical state.

#### Reports:

No. 1-2 in previous edition.

3. Transient Boundary-Layer Flows in Combustion Environments, by S. W. Kang, et al, pres 19th AIAA Aerospace Sci Meeting.
4. Interaction of Moderately Dense Particle Concentrations in Turbulent Flow, by Alfred C. Buckingham et al, pres AIAA 19th Aerospace Sci Meeting.
5. Additive Erosion Reduction Influences in the Turbulent Boundary Layer, by Alfred C. Buckingham, Proc 81 JANNAF Propulsion Meeting.

### 16002 AN INVESTIGATION OF THE ELECTROCHEMICAL BEHAVIOR OF GRAPHITE FIBER-POLYMER COMPOSITES

G. E. Stoner  
G. I. Cahen  
University of Virginia  
SI: MERADCOM SC: CSL, CERL, MERADCOM

This investigation has primarily dealt with the mechanical and voltage stability of composite electrodes in particular aqueous environments and under controlled electrochemical conditions. The potential for these materials in electrochemical applications is for reaching. A composite containing graphite fiber which is oriented perpendicularly to the solution interface offers a number of interesting possibilities. The fabrication of such composites is sufficiently advanced so that the fiber loading and therefore the fiber separation can be fairly well controlled. Such a configuration would be ideal for selectively electroplating a catalyst material onto the graphite fiber, thus controlling both the catalyst size and separation. A new electrochemical cell is being designed which will allow the electrode to be readily removed and

examined in the scannery electron microscope in a routine manner. Research is focussing in more detail on where the electrochemically active and inactive parts of the graphite fibers are located.

#### 17082 ELECTROCHEMICAL CONDITIONS WITHIN STRESS CORROSION CRACKS

M. Marek

Georgia Institute of Technology

SL: AMMRC SC: MERADCOM, MICOM, FCSCWSL

The objective of the program in the first year is to develop improved techniques of determination of electrochemical conditions within stress corrosion cracks. The parameters to be determined include acidity (pH), chloride ion concentration, concentration of cations (ions of alloying elements), and electrode potential. Standard techniques of analysis of corrosion products and fracture surfaces are expected to be used in the later phases of the program. Techniques to be used in the research include potential measurements with an implanted reference probe, and pH and potential measurements with probes positioned along the crack trace on the side of the specimen. It is recognized that the latter technique does not allow measurement of pH near the tip of the crack; however, the results provide reference points against which data from other tests can be checked. The preliminary data on the potential variation along the crack length are consistent with the results from the implanted electrode. Together they show the potential drop from the mouth of the crack to the leading edge which will be possible to correlate with the theoretical predictions.

#### 17415 AN INVESTIGATION OF THE MOISTURE SENSITIVITY OF EPOXY RESIN-GRAPHITE FIBER COMPOSITES USING BROADLINE NMR, IR AND CHEMICAL CHARACTERIZATION STUDIES

J. D. Memory

R. E. Fornes

R. D. Gilbert

North Carolina State University

SL: LANGLEY, AMMRC SC: FCSCWSL, MERADCOM

The objective of the investigation is to study the interaction of moisture with an epoxy resin-graphite

fiber composite system. The program will include: (1) Determination of soluble species by extraction with solvents. (2) Determination of amine/hydroxyl group ratios and changes in carbonyl groups using FTIR. (3) Determination of uptake of moisture using gravimetric techniques. (4) Determination of the degree of cure of the epoxy matrix using high performance liquid chromatography. (5) Determination of sensitivity to moisture using FTIR, broadline NMR, and mechanical testing.

#### Reports:

1. Temperature Dependence of Broadline NMR Spectra of Water-Soaked Epoxy-Graphite Composites, by David Lawing et al.

#### 17421 INTERACTIONS BETWEEN EROSION AND CORROSION OF METALS AND ALLOYS AT ELEVATED TEMPERATURES

F. S. Pettit

N. Birks

University of Pittsburgh

SL: AMMRC SC: LEWIS, EUSTIS, TARADCOM

The objective of this research is to study the mechanism of erosion-corrosion of metals and alloys under conditions of high gas temperature and steady-state gas flow, with and without particulate. The research will include: (1) Development of an erosion/corrosion apparatus. (2) Erosion-oxidation studies to determine the interrelated effects of alloy composition, temperature, environmental gas composition, and particulate size, morphology, flow and impact characteristics on the interaction between the influence of gas phase thermodynamics and kinetics and particulate damage in erosion/corrosion. (3) Definition of erosion/corrosion mechanisms and the regimes under which each mechanism is prevalent.

#### 17799 ENVIRONMENTAL EFFECTS UPON TIME-DEPENDENT FAILURE OF NON-OXIDE CERAMICS

Richard E. Tressler

Pennsylvania State University

SC: MERADCOM, TARADCOM, LEWIS

The objective of the investigation is to study the environmental effects on the time dependent



fracture on non-oxide ceramics. The program will include: (1) Quantitative analysis of the mechanisms of oxygen penetration and sintering aid redistribution during thermal oxidation of SiC and Si<sub>3</sub>N<sub>4</sub>, and of the effects of impurities (and spatial distribution) on the kinetics of reactions using SIPS, SIMS, ellipsometry, SEM and TEM. (2) Measurement of mechanical properties at various stages of the reactions, e. g. double torsion crack growth rate, dynamic fatigue, static loading, notched beam tests, hot hardness. (3) Analysis of time-dependent reactions and mechanical behavior to form a predictive model for time-dependent failure in service.

#### 17982 EFFECT OF MOISTURE AND TEMPERATURE ON POLYMER MATRIX COMPOSITES

A. A. Fahmy  
J. C. Hurt  
North Carolina State University

Earlier work was concerned with the effects of stress on the diffusion coefficient of small molecule solutes in glassy polymers. It was theoretically predicted and experimentally verified that the existence of a tensile stress in the polymer would increase (and a compressive stress would decrease) the diffusion coefficient. However, the amount of solute and its distribution as described by the concentration-penetration profiles depend on the equilibrium concentration of solute in the polymer as well as the diffusion coefficient. Current research efforts are mainly concerned with the effect of stress on equilibrium concentration of solutes in glassy polymers.

#### 18043 TRANSPORT OF ANION IMPURITIES THROUGH OXIDES

J. Bruce Wagner  
Arizona State University  
SL: AMMRC, LEWIS

The objective of the research is to study foreign ion diffusion mechanisms in oxides, with special attention to the cases of sulfur diffusion in yttria- and calcia-stabilized zirconia and nickel. The research will include: (1) The dual diffusion mechanisms of

sulfur in oxide single crystals will be examined for the case of complex yttria- and calcia-stabilized zirconia in order to determine if the model of ionic transport which was developed can be extended to this class of material. (2) The kinetics of nickel degradation in hot SO<sub>2</sub> and SO<sub>3</sub> will be studied to gain an understanding of the effects of mixtures of gases on the mechanisms of sulfidation of nickel.

#### 18205 DEVELOPMENT OF A SCANNING DEVICE FOR CORROSION STUDIES

H. S. Isaacs  
Brookhaven National Laboratory  
SL: AMMRC, FCSCWSL

The objective of this research is to transfer technology originating with the concepts developed under ARO funding of Project No. 9075-MC, "Role of Surface Structure and Applied Stress in Corrosion," by development of two advanced corrodscopes utilizing both alternating and direct current scanning techniques to nondestructively evaluate the propensity for general and localized corrosion of bare and coated metallic surfaces. The details of the research are detailed below: (1) *Instrumentation*: The system proposed will consist of two scanning devices for scanning planar and cylindrical specimens. The scanning will be controlled by a desk top computer that will also record information from a data acquisition system. The data will be permanently stored on either cassettes or floppy discs. The scanning will either determine the variations in d. c. potential above the surface or the variations in a. c. impedance. The d. c. measurements will be made with an electrometer and, if required, a potentiostat. The a. c. impedance will be determined with a lock-in amplifier at a frequency set by a signal generator. The data display will be initially on the CRO screen of the computer and then transferred to the X-Y plotter for a permanent record. (2) *Software*: The programming of the computer will be divided into three sections, equipment control, data acquisition, data manipulation and presentation. The control of the equipment will be programmed to incorporate the user's scan rate, step size, and area to be covered within the limitations set by the instrumentation or data acquisition rate. The software for the data acquisition will incorporate the control information and data storage procedures with

required buffers, and writing permanent files. The data presentation will be of two forms, either as three dimensional surfaces or contour mapping. In order to plot the data, the software for the three dimensional plotting will have to be developed. All or part of the data for the area scanned may be displayed. The coordinates of the areas of maximum or minimum response will be determined and may be chosen for rescanning. (3) *Corrosion Studies*: During the development and setting up of the instruments, measurements will be conducted on a series of samples of interest to the U. S. Army research and development programs. The sample may include metal coating, organic coating, bare metal surfaces or welded structures prepared on both planar and cylindrical specimens.

## B. MECHANICAL BEHAVIOR

### 14889 FAILURE PREDICTION IN GLASS

R. H. Doremus  
Rensselaer Polytechnic Institute  
SL: CSTA SC: AMMRC, ETDL, MERADCOM

A large number of additional measurements of delayed failure times for Pyrex glass rods under widely differing stresses have been made. The results were analyzed statistically and significant differences were found between two different sets of apparatus. The reasons for this difference are being analyzed with direct measurements of strain on samples. A set of data taken on a particular apparatus by the same workers has proven to be especially useful for analysis. In a direct statistical test of various relations between log failure time and applied stress it was found that the equation  $\log t = K_1/S + K_2(1)$  where  $K_1$  and  $K_2$  are constants fit better (high correlation coefficient) than other proposed equations. This fit was confirmed by the dependence of the spread (standard deviation) in failure times on stress—the spread increases at lower applied stresses, and the amount of the increase is close to that expected. Long term measurements also are better predicted by Eq. 1 than other equations. It was concluded that Eq. 1 provides the best fit to experimental static fatigue data on Pyrex glass, and should be used for extrapolations of fatigue behavior of Pyrex and other silicate glasses. In a statistical analysis of the fit of fracture data to the normal and Weibull distributions, it was

concluded that the normal distribution provides a better fit, and the "best" parameters for the normal distribution are easier to calculate than for the Weibull.

#### Reports:

- No. 1-5 in previous editions.
- 6. Static fatigue of Glass: Functional Dependence of Failure Time on Stress, by G. S. Friedman et al, *J Mater Sci.*
- 7. Fracture and Fatigue of Glass, by Robert H. Doremus, *Glass III*, ed by R. H. Doremus et al.

### 15098 CHARACTERIZATION OF MECHANICAL, THERMAL AND WEAR PROPERTIES OF Ti-RICH METALLIC GLASSES

B. C. Giessen  
Northeastern University  
SL: CERL, AMMRC SC: MERADCOM, ERADCOM, BWL

*Design and Construction of Wear Test Unit*: The wear test unit has been designed and constructed. This unit allows the measurement under controlled atmosphere conditions of the wear and friction coefficients of foil samples produced by rapid quenching. *Development of Method for Measuring E on Short Samples*: A new method has been tested for measuring the Young's modulus E of short glass samples. *Corrosion Tests*: Dynamic voltammetric corrosion tests were carried out on Ti-Ni-Si alloy glasses in neutral-to-concentrated acidic chloride solutions. Preliminary results showed levels of corrosion resistance comparable to those measured on melt-spun "Metglas" type alloys containing Cr. *Relationship of Microhardness and Elasticity*: A comprehensive study has been made of the Young's modulus E and the Vicker's microhardness  $H_v$  for many transition-metal glasses belonging to three different families. *Compositional Dependence of E,  $H_v$ , and  $T_c$  of Glassy Ti Alloys*: An extensive study of the compositional dependence of the inter-related thermal-elastic-plastic properties of glassy alloys with compositions  $(Ti_xNi_{1-x})_{90}Si_{10}$  has been carried out. In parallel with this work, the effect of different metals M on these properties has been studied in glassy alloys with the composition  $Ti_{60}M_{30}Si_{10}$  where M = Fe, Co, Ni, Cu. *Theoretical Programs*: Effects were made to analyze some of the observed properties of glassy metals theoretically, using a linear lattice model as a

simplifying approach to obtain a calculable situation. Suitable inter-atomic potentials and the formulations of discontinuum Born theory were employed. Specifically, the composition dependence of the elastic and thermal properties, radial distribution functions, and densities have been studied. Further, an attempt was made to explain theoretically the maxima of the colligative properties mentioned above.

*Reports:*

No. 1-2 in previous edition.

**15112 MECHANISM OF RECOVERY IN PLASTIC DEFORMATION AND CREEP**

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SC: MERADCOM, LEWIS

The high resolution electron microscopy of the dislocation and "debris" content of sub-grain boundaries carried out both at M.I.T. by means of the weak beam technique and at the Argonne National Laboratory on the high voltage electron microscope have enabled the development of more precise models for boundary mobility, and the competing processes of both thermal and dislocation dynamical boundary cleanup, as well as new "debris" accumulation. Based on these, two complementary models have been developed. A series of creep experiments on copper single crystals utilizing the well known dislocation etching technique of Livingston is being used to follow the subgrain structure transients upon stress change to decide on the rate of attainment of a new steady state upon stress change. In addition, the nature of both stress increase and stress decrease (dip test) transients in both copper and nickel single crystals are being investigated with a goal associating them with mobile dislocation density build-up and decay and subgrain flexing and straightening. Additional in-situ annealing experiments have been conducted at the Argonne National Laboratory on the stages of boundary clean-up in recovery on previously deformed and crept nickel single crystals. These results together with the previous annealing experiments are now being quantitatively analyzed to ascertain the kinetics of disappearance of the various redundant defect structures leaving behind only the geometrically necessary dislocations.

**15299 MORPHOLOGY AND KINETICS OF STRAIN-INDUCED CRYSTALLIZATION OF POLYMERS DURING FLOW**

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University of Michigan  
SL: AMMRC, CSL SC: MERADCOM

The melting behavior of shear-crystallized polyethylene was examined carefully in a DSC under unrestrained as well as under properly restrained conditions. The polymer showed two melting temperatures: a lower  $T_{m,l}$  occurring at about 132C was established as the  $T_{m,TIC}$  of thermally-induced crystallinities and a higher  $T_{m,b}$  occurring between 141 and 148C was established as the  $T_{m,SIC}$  of strain-induced crystallinities formed during shearing.  $T_{m,SIC}$  increases with increasing shearing temperature (134-142 C) and with increasing shear rate ( $1.32-7.48 \text{ sec}^{-1}$ ). The shear rate dependency indicates that the  $T_m$  increase in shear-crystallized polyethylene, as in the case of other strain-crystallized polymers, is also entropic in origin. From the measured increases, the degree of melt entropy reduction  $\Delta S'$  and the coil deformation reached just prior to strain-induced crystallization were estimated to be  $1-1.5 \times 10^5 \text{ ergs/cm}^3$  - degrees K and 25-50%. These results are consistent with morphological findings which suggest very limited chain extension prior to strain-induced crystallization.

**15628 CYCLIC DEFORMATION AND FATIGUE OF PRECIPITATE-HARDENED SINGLE CRYSTALS**

Campbell Laird  
University of Pennsylvania  
SC: AMMRC, MERADCOM

Persistent slip band (PSB) behavior has been observed in detail by interferometric techniques at two strain amplitudes, one near the low strain end of the plateau and one near the high strain end. Unlike pure metals the gauge length is not saturated with PSB's at the upper end of the plateau; nevertheless the volume fraction of PSB's increases by a large fraction as the strain increases in the plateau. The first PSB's are observed to form just before the peak and are clearly observed at the moment of formation on the specimen side oriented

at 90% to the primary Burgers vector. This indicates that the formation of the PSB's coincides with the operation of some slip by dislocations having a Burgers vector different from, but coplanar with, the primary Burgers vector. The strain concentrated in the PSB's varies by a large amount, from several percent to many tens of percent. These strains are calculated from the heights of the slip offsets (associated with primary dislocations) and the widths of the PSB's as measured by TEM in the bulk of the sample. Measurements have been made of PSB behavior during the softening that follows the peak hardening. This has been associated with increasing amounts of strain localization (by factors of two or more) but the density of the active PSB's does not decrease as softening develops, i.e., the histogram of slip offsets in the PSB's simply moves to higher values of offset with more cycling. The fatal crack is observed to develop in the PSB (usually a group of closely spaced PSB's) with the highest strain concentration; such PSB's reveal themselves at peak hardening and one can select with great confidence at that point the macro-PSB in which the crack will form.

#### Reports:

No. 1-4 in previous editions.

5. Cyclic Deformation, Fatigue Crack Nucleation and Propagation in Metals and Alloys, by Campbell Laird.
6. Cyclic Deformation of Polycrystalline Pure Metals and of Precipitation Strengthened Alloys, by A. S. Cheng et al.
7. Cyclic Deformation in Aluminum - 4 Wt% Copper Alloy Single Crystals Containing Coherent  $\theta''$  Precipitates, Part II, Dislocation Microstructures, by Jia-Kuen Lee et al.
8. Cyclic Deformation in Aluminum - 4 Wt% Copper Alloy Single Crystals Containing Coherent  $\theta''$  Precipitates, part I, Cyclic Stress-Strain Response, by Jia-Kuen Lee et al.

### 15643 INTERDISCIPLINARY RESEARCH PROJECT ON SLIDING WEAR

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SL: AMMRC, ARRADCOM SC: MERADCOM,  
ARRADCOM

Investigations have continued on the generation and characterization of wear debris produced during lubricated wear and the observation of the wear-in process with *in situ* wear experiments in the scanning electron microscope. Lubricated wear experiments have involved block on ring rubbing contact using three copper binary alloys: Cu-3.5Al,

Cu-7Al and Cu-15Zn. The lubricant has been a 1% solution of stearic acid in dodecane. Recent experiments have also included a diester lubricant to preclude soap formation in the lubrication process. The mathematical modeling of delamination type wear has continued with analysis beyond the fully elastic model. At the outset, it was recognized that the elastic model was an over simplified approach and that plasticity was an essential element. Accordingly, a preliminary elastic-plastic model has been developed. Wear tests on austenitic stainless steels have been completed. The latest experiments have included more stable austenite alloys as well as some self-mated tests. The results on 304, 305, 316, 332, and 310 alloys confirm the importance of strain-induced martensite as found by Hsu. The work on Cu-Ni alloys has now been completed. Initially, it was expected that stacking fault energy and hardness would be important variables. Subsequently, what seemed to be changes in oxidation behavior has been observed which raised the possibility of stress-corrosion cracking as a contributor. Intensive study on many Cu-Ni alloy samples showed that higher nickel content correlated with a greater tendency for material transfer. As a result, this project has increasingly focused on details of the transfer process.

#### Reports:

No. 1-7 in previous editions.

### 15689 MICROSTRUCTURE AND FRACTURE TOUGHNESS OF AN HSLA STEEL

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SL: AMMRC, BWL SC: ARRADCOM, MERADCOM

The objective of this program is to develop an understanding of the effects of variation in microstructure on the fracture toughness of an HSLA steel. Charpy V-notch impact tests have been performed on specimens with each heat treatment. Scanning-electron-microscope examination of the fracture surfaces of room-temperature Charpy specimens is in progress. Specimens tested in the as-rolled-and-aged condition have been examined, as well as specimens which had been given heat treatments. The fracture surfaces of those Charpy specimens were very



similar. All had dimpled surfaces characteristic of ductile fracture. The mechanism probably was nucleation of voids at second-phase particles, followed by void growth, coalescence, and junction with the crack tip. The second-phase particles involved in void nucleation could include carbides and sulfides; islands of martensite mixed with austenite also may be involved. Deep, narrow pits perpendicular to the fracture surfaces and long, shallow grooves on the fracture surfaces suggest that manganese sulfide stringers act as void-nucleation sites. Smaller, equiaxed sulfide inclusions have been identified in some of the dimples, so they, too, nucleate voids. Plans are being made to expand the investigation of the microstructure by using various etchants, combined with SEM examination, to establish the number and type of inclusions present, whether martensite-austenite islands exist in any of the microstructures, and the role of the above in fracture.

#### Reports:

1. J Integral Analysis of Stable Crack Growth, by I. H. Lin et al, *Intl J Fracture* 16, R11 (1980). (see page 5)
2. Plastic Instability in Uniaxial Tension Tests, by Ing-Ho Lin et al, *Acta Met* 29, 819 (1981).
3. Crack Arrest in a Wire-reinforced Polymer Composite, by C. R. Barnes et al, *Mater Sci Eng* 43, 261 (1980).
4. Key Curve Analysis of Crack-Growth-Resistance Curves, by I. H. Lin et al, *Intl J Fracture*.
5. RESPONSE: Discussion of "J Integral Analysis of Stable Crack Growth", by S. J. Burns, *Intl J Fracture* 16, R157 (1980). AD A091 397
6. Stress Intensity Factors For Non-Uniform Applied Stresses, by A. R. Rosenfield, *Eng Fracture Mech* 13, 1031 (1980). AD A092 675
7. Mixed-Mode Crack Growth Using the Compact Specimen, by M. T. Miglin et al.

### 16123 MICROMECHANICAL PROPERTIES OF CRAZES

E. J. Kramer  
Cornell University  
SC: AMMRC, MERADCOM

A new method of producing sharp cracks in thin polymer films has been developed. The sharp cracks have made it possible to grow environmental crazes and plane stress plastic zones from crack tips in PC films. The morphology, microstructure and fracture behavior of crazes produced by sequentially applying tensile strains orthogonal to

each other were investigated. Under certain conditions sets of intersecting crazes were produced. It was found that: (1) When two crazes intersect, the fibril volume fraction in the intersection is approximately the product of the fibril volume fractions of the two crazes, and is therefore much lower than the normal craze fibril volume fraction. (2) The product law for the fibril volume fraction follows directly from the surface drawing mechanism for craze thickening. (3) The lower fibril volume fraction results in higher fibril stresses in the intersection giving rise to more rapid fibril breakdown there to form voids. These can act as sites from which cracks develop prematurely. (4) Both craze intersection and fibril breakdown in the intersection occur more frequently at higher applied strains (and stresses). (5) Given the evidence that craze intersections can act as efficient nuclei of cracks, care should be taken to avoid stress histories which can result in formation of intersecting crazes.

#### Reports:

- No. 1-4 in previous editions.
5. Film Thickness Effects on Craze Micromechanics, by Terence Chan et al, *J Mater Sci* 16, 676 (1981).
  6. Craze Intersections in Biaxially-Stressed Polystyrene, by Paul S. King et al, *J Mater Sci*.
  7. Plane Stress Deformation Zones at Crack Tips in Polycarbonate, by Athene M. Donald et al, *J Mater Sci*.
  8. Micromechanics and Kinetics of Deformation Zones at Crack Tips in Polycarbonate, by Athene M. Donald et al, *J Mater Sci*.
  9. Effect of Molecular Entanglements on Craze Microstructure in Glassy Polymers, by Athene M. Donald et al, *J Polymer Sci-Polymer Phys*.
  10. The Effect of Strain History on Craze Microstructure, by Athene M. Donald et al.

### 16143 EROSION OF DUCTILE MATERIALS

J. W. Edington  
University of Delaware  
SL: AMMRC SC: FCSCWSL, MICOM, LEWIS

Examination of eroded surfaces has demonstrated for the first time that stable hills and valleys form on erosion of electropolished surfaces of single crystals of copper and polycrystalline, fully annealed aluminum. Material loss processes in steady state erosion involve micromachining from the sides of the hills and flaking by subsurface cracking from the bottom of the valleys. The latter mechanism is dominant in copper but not aluminum after the early stages of steady state erosion. Scanning

electron microscopy studies of metallographic sections of eroded material have demonstrated extensive embedding of erodant in the valleys in copper but not aluminum. Heavy sub-surface cracking is observed in both materials. Large voids are observed in aluminum. There is evidence of extensive material flow in the bottoms of the valleys in both materials. Examination of erosion debris by transmission electron microscopy has shown clear evidence for recrystallization and surface heating during steady state erosion. In addition, small voids and cracks, not associated with hard particles, were observed. These are thought to arise directly from the plastic deformation process associated with the multiple impact of particles. To obtain information on the temperature rise during erosion a series of aluminum-gallium alloys with melting temperatures of 500, 400, 300, 200 and 100C and pure gallium, m.pt. 27C, were eroded by glass spheres in a whirling arm rig under vacuum at 900 ft. s<sup>-1</sup>. The only evidence for melting was found in the pure gallium. Therefore the temperature rise under these conditions appeared to be between 27 and 100C. Copper single crystals were chosen as a model material for erosion studies, while the gallium alloys were being produced, due to their simple and easily characterized microstructure. Any changes in the structure due to erosion were then easily determined.

#### Reports:

- No. 1 in previous edition.
2. Mechanism of Erosive Wear for 90 Degree Impact on Copper and Iron Targets, by R. Brown et al, pres 3rd Intl Conf on Wear.
3. Erosion of Copper Single Crystals Under Conditions of 90 Degree Impact, by R. Brown et al, *Wear*.

### 16227 EFFECTS OF SHOCK PULSE VARIABLES ON SHOCK STRENGTHENING OF METALS

D. E. Mikkalo

Michigan Technological University

SL: ARRADCOM, SC: AMMRC, MICOM  
MERADCOM, BRL

The analysis of the effects of initial grain size, shocking temperature, and multiple shocking on the substructure and properties of Cu-8.7Ge has been completed. Also, the room temperature shock hardening response of pure Cu has been compared

to that for Cu-8.7Ge. Increasing the grain size of Cu-8.7Ge decreases the plastic strain contribution from twinning, however, the general variation of hardening with pulse duration is unchanged. That is, a maximum in hardening occurs followed by a minimum and a subsequent increase to saturation at long pulse durations. The loss of strain from twinning with the larger grain size is offset by the generation of the dislocation substructure within shorter times. Lowering the deformation temperature to 166K also caused a small decrease in the strain contribution from twinning. Again, this was offset by a more rapid development of the dislocation substructure. Multiple shocking has shown twinning effects to be cumulative and predictable from single shock results, however the resulting dislocation density is greater than expected. The hardening for all of these cases can be described in a semi-quantitative manner with a modified Hall-Petch expression. Detailed analysis of all of these results has established that the strength coefficients for twin boundaries are ~ 1% of those for grain boundaries in the alloy. Further evidence has accumulated showing that deformation twins form at least 1000 times faster than previously thought. The shock hardening response of pure Cu is similar to that for Cu-8.7Ge except that the defect densities are smaller and saturation occurs within shorter times.

#### Reports:

1. Short Duration Shock Pulses as a Tool to Study the Time Dependence of Plastic Deformation, by R. N. Wright et al, *Proc of Intl Conf on Met Effects of High Strain-Rate Deformation and Fabrication*.

### 16425 THE CONTROL OF POLYMER MECHANICAL BEHAVIOR BY SURFACE MODIFICATION

I. J. Broutman

Illinois Institute of Technology

SL: CSL, AMMRC SC: MERADCOM

Prior studies have concentrated on the mechanical behavior of surface modified high impact polystyrene (HIPS) and ABS polymers. The results obtained with these two polymers have been analyzed with respect to the development of a theory to explain the embrittlement phenomenon observed when brittle compatible polymers were laminated to the surface of the ductile polymer. In addition, a polycarbonate polymer has been added

to the ductile materials to be studied. This material has been chosen because of its different deformation mechanism in comparison to the rubber modified polymers and also because of its sensitivity to surface embrittlement. In order to simulate the embrittlement phenomenon, an epoxy resin layer has been cast onto the polycarbonate surface. Polycarbonate samples are coated with epoxy layers of different thickness, and it is seen that as the layer thickness increases, the strain to failure of the sample decreases. In order to determine more quantitatively the role of crack velocity or kinetic energy, a new approach is being evaluated. In other words, one hypothesis accounting for the effect of surface layer thickness on embrittlement, suggests that as the brittle surface layer thickness increases, cracks initiated in this layer can reach higher velocities at the time they reach the brittle-ductile boundary. Thus, as the thickness increases, the embrittlement should become more severe. A test specimen and method have been designed to determine a critical crack velocity required to initiate and propagate a crack in the ductile polymer.

#### Reports:

No. 1 in previous edition.

### 16556 DYNAMIC PROPERTIES OF FIBER REINFORCED CONCRETE UNDER IMPACT LOADING

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A. E. Naaman

University of Illinois at Chicago Circle

SL: CERL SC: WES, CRREL, MERADCOM, CERL

An experimental program dealing with the pull-out response of fibers from a portland cement mortar matrix at various loading velocities is underway. Three types of fibers, namely, steel, glass and polypropylene are being used. A range of loading velocities from  $4.2 \times 10^{-3}$  cm/sec to 300 cm/sec is being explored. A special experimental procedure has been developed. It allows the pulling out of a group of fibers simultaneously hence reducing the scatter in the results. The effects of loading velocity (strain rate) on the load-time response of fibers, their equivalent bond strength and their energy absorption to failure or to complete pull-out were analyzed. Also, the limitation of the testing procedure and its influence on the reliability of the

results has been studied. Because of the difficulties encountered in eliminating the influence of parasitic and interactive effects between specimen and testing system at impact, and because such effects tend to blur the initial portion of the load-time response, a modification of the testing procedures is currently being undertaken. It includes adding instrumented gages to the anvils or support system in order to record the reaction versus time response as well as the applied load versus time obtained from the tup.

### 16667 ORDERING AND PLASTIC DEFORMATION IN ZIRCONIA-BASED CERAMICS

A. H. Heuer

T. E. Mitchell

Case Western Reserve University

SL: LEWIS, AMMRC SC: MERADCOM, TARADCOM

The kinetics, morphology and crystallography of precipitation reactions in Ca-stabilized  $ZrO_2$  have been determined. In hypo-eutectoid compositions ( $<15\%$  CaO), tetragonal zirconia precipitates form in the cubic solid solution during isothermal aging heat treatments between 1000 and 1400°C. The precipitation process involves nucleation and diffusional coarsening, and the rate of particle coarsening follows cubic kinetics which is typical of a diffusional coarsening process that is driven by the minimization of total interfacial energy but rate limited by diffusion. Major attention is now being given to hypo- and hypereutectic compositions, which have been heat-treated to precipitate tetragonal  $ZrO_2$  and  $CaZr_2O_6$ , respectively, and which will be deformed at temperatures within the respective two-phase fields. In the first experiments of this kind, the CRSS appears to be increased by a factor of two, compared to a single phase crystal of the same composition, suggesting that precipitation hardening may be particularly potent in  $ZrO_2$ -based systems.

### 16810 DUCTILE VS BRITTLE FRACTURES

R. Thomson

B. J. Hockey

National Bureau of Standards

SC: AMMRC, MICOM, FCSCWSL

A general picture of how a sharp crack is shielded by a screening distribution of dislocations has been

developed. A theorem has been developed which allows the force on the crack and its screening cloud to be calculated, and which shows that the total force on a crack plus its screening cloud is balanced by the rate of work done in creating a wake of debris behind the moving configuration. The theory also shows that the screening cloud is balanced by a crack opening displacement whose magnitude is equal to the total burgers vector in the cloud. A paper is being prepared on these results.

#### **16850 SUPERPLASTIC BONDING AND ULTRAHIGH CARBON STEEL LAMINATED COMPOSITES**

O. D. Sherby  
Stanford University

SL: BWL, AMMRC SC: MERADCOM, TARADCOM

This program is concerned with the synthesis and preparation of ultrahigh carbon (UHC) steel laminated composites and a study of the mechanical behavior of these ferrous laminated composites. Efforts have been concentrated on three aspects of the UHC steel laminated composites. The first area centers on an analysis of the high temperature mechanical behavior of UHC (1.5%C) steel-I. F. iron laminated composites. The second area centers on the synthesis, preparation and properties of a UHC (1.22%C) steel-mild steel laminated composite. The third area related to some preliminary selective heat treatment studies on the microstructure of the UHC (1.22%C) steel-mild steel composites.

#### **17401 STUDY OF THE EFFECTS OF METALLURGICAL FACTORS CONTROLLING THE GROWTH OF FATIGUE MICROCRACKS**

James Lankford  
Southwest Research Institute

SL: BWL SC: AMMRC, FCSCWSL, MERADCOM

The objective of the research will be to determine the metallurgical bases for deviations in fatigue microcrack behavior from the predicted on the basis of large crack macroscopic fracture principles. The research to be performed will

include studies of the effects of the following factors on the growth of small fatigue cracks in low carbon ferritic steel, an iron-silicon alloy, 7075 aluminum alloy, and a copper-aluminum alloy: Stacking-fault energy/slip character, grain size, crack size, and plastic zone size.

#### **17567 THEORETICAL INVESTIGATION OF CREEP FRACTURE IN METALLIC MATERIALS**

V. Vitek  
University of Pennsylvania  
SC: AMMRC, BWL

The research objective is to investigate the following major problems in the development of the theory of creep fracture: (1) Interaction between diffusional cavity growth, creep plasticity inside the grains, and grain boundary sliding. (2) Effects of surface diffusion. (3) Mechanisms of cavity nucleation and their effect on creep fracture. (4) Investigation of the validity of the assumption that grain boundaries can act as ideal sources of vacancies. The research will include a study of the diffusional growth of cavities in a polycrystal when the grains undergo power law creep. Both uniformly distributed cavities and cavities ahead of a crack will be studied, and the effects of surface diffusion and cavity nucleation will also be taken into account. The effects of parameters such as the distribution of carbides and inclusions, grain size, creep strength, etc. will also be considered.

#### **17653 CARBIDE FORMATION AND FRACTURE OF HIGH CARBON STEEL**

G. Krauss  
Colorado School of Mines  
SL: AMMRC SC: FCSCWSL, MERADCOM

The research objective is to study carbide formation during austenitizing, cooling, and tempering in 52100 steels, Fe-Cr-C, and Fe-Mo-C alloys as related to fracture toughness. 52100 steels with various P additions will be used to test and extend the observation that phosphorous strongly enhances the rate of cementite formation. The



52100 steels and other alloys will be used to characterize the kinetics and mechanisms of carbide grain boundary growth by light microscopy, transmission and scanning electron microscopy, and Auger electron spectroscopy. Fracture transitions as related to tempering will also be studied by electron microscopy and other techniques.

#### 17752 AGE-HARDENING OF BINARY, MARTENSITIC IRON-NICKEL ALLOYS

W. C. Leslie

University of Michigan

SL: BRL SC: FCSCWSL, MERADCOM, BWL

The objective of the research is to study the mechanism for unexpected age-hardening in binary martensitic Fe-Ni alloys. The research will include studies of the mechanical and magnetic properties of the aged alloys, and will focus on the size, shape, structure, number density, and nucleation sites of the hardening agent. The results will be used to clarify the form of the Fe-Ni phase diagram at low temperatures and will be correlated with structures and properties observed by other investigators in meteorites and in irradiated alloys.

#### 17887 INVESTIGATION OF MATERIAL FACTORS IN THE ADIABATIC SHEARING OF STEELS

H. C. Rogers

Drexel University

SL: FCSCWSL, AMMRC SC: BWL

The research objective is to gain improved understanding of the nature of adiabatic shear in ferrous alloys, principally steels. The study will include the investigation of the roles of composition, microstructure, and transformation characteristics on the tendency to produce transformed adiabatic shear bands during dynamic deformation. Thermal and mechanical characteristics of the alloys will also be varied in order to incorporate these into a simplified model of the adiabatic shear process. Detailed studies of transformed shear band nucleation and growth as related to heat treatment will also be carried out.

#### 18053 MARTENSITIC TRANSFORMATION IN IRON ALLOYS

C. M. Wayman

University of Illinois at Urbana

SL: CERL SC: LCWSL, MERADCOM, AMMRC, BRL

The objectives of this research are a continuation of the investigation of how to control the growth of lath martensite to create a directionally aligned, fibrous martensitic microstructure in steels, and to obtain the subsequent mechanical properties. Utilizing an Fe-Ni-Mn alloy, an aligned martensitic structure will be attained via various treatments discovered in earlier work and based on isothermal lath martensite. The mechanical properties will be determined as a function of treatment, with accompanying detailed analysis of the Fe-Ni-Mn lath martensite morphology via transmission electron microscopy.

#### 18184 A STUDY INTO THE MECHANISMS FOR THE ELECTROPLASTIC EFFECT IN METALS AND ITS APPLICATION IN METALWORKING

Hans Conrad

North Carolina State University

SL: BRL SC: FCSCWSL, LCWSL, MERADCOM, BWL

The objective of this research is to experimentally investigate the electroplastic effect in metals in order to obtain a fundamental understanding of the effect and evaluate its application on metal working processes. The research program will include: (1) Evaluation of the electroplastic effect over the temperature range of 77-130K for the following metals: Cu, Ni, and Al (fcc) and Nb, Nb-Mo alloys and W (bcc). These metals give a difference in valence electrons/atom. Tests will be conducted on both polycrystalline and single crystal specimens. (2) A comparison of the electroplastic effect with the normal to superconducting effect will be carried out on the Nb and Nb-Mo alloy single crystal specimens. These tests should provide greater insight into the nature of the electron-dislocation interactions. (3) An investigation of the potential of the electroplastic effect in metal working applications by drawing polycrystalline wires of the metals listed in (1) above while applying high current density pulses during the drawing operation.

**18487 STUDIES IN DEFORMATION AND FRACTURE**

C. R. Manning, Jr.  
G. Mayer  
North Carolina State University

The objective of the research is to clarify the fatigue and corrosion-fatigue behavior of metal-matrix composite materials. Measurements will be made of crack propagation under fatigue and corrosion-fatigue conditions in aluminum/graphite, aluminum/aluminum oxide, and aluminum/silicon carbide materials. Fracture toughness analysis will be performed from the foregoing data. Fracture surfaces will be analyzed by optical and scanning electron microscopy, and coupled with the fracture analysis, will lead to determination of the dominant failure modes in metal-matrix composites undergoing cyclic loading in corrosive environments.

**C. SYNTHESIS AND PROCESSING****14858 REFINING BY SOLIDIFICATION**

M. C. Flemings  
D. R. Poirier  
Massachusetts Institute of Technology  
SL: AMMRC, FCSCWSL SC: DARCOM,  
MERADCOM

Three major papers have been written so far on the results of the work. The first of these papers describes an invention of refining by squeezing during melting. The second is the patent which has been applied for, for this process. The process is vastly more efficient than simply refining by removal of the liquid isothermally. The third paper above describes work on using the process to strengthen 7000 series alloys. This work was done on relatively small samples but results of that work showed that strengthening by fractional melting produces 7000 series alloys with room temperature tensile properties equivalent to or exceeding the best produced by any other process, including consolidation of splat-cooled powders. A related study to the foregoing is concerned with effects of refining efficiency of applied forces, thermal changes, and other process variables, using aluminum-copper alloys.

*Reports:*  
No. 1-3 in previous edition.

**14868 MARTENSITIC TRANSFORMATIONS IN IRON ALLOYS**

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University of Illinois at Urbana  
SL: AMMRC, MERADCOM SC: TARADCOM, BRL

Further experiments have been conducted with alloys near the composition Fe-20Ni-5Mn (wt%) to determine fundamental characteristics of the austenite-martensite transformation in these materials and to explore the feasibility of the production of an aligned martensitic microstructure with superior mechanical properties. The martensite found in Fe-20Ni-5Mn is of the lath type, typical of that found in low carbon and HSLA steels, but in the former case the transformation is isothermal with C-curve kinetics and the  $M_s$  temperature is below room temperature. Morphological studies have been made on alloys consisting of Fe-20Ni-4.9 Mn, Fe-19.8 Ni-5.3Mn and Fe-20.2Ni-5.7Mn, and preliminary results indicate that even over this rather narrow variation in composition, the martensite morphology (i.e., lath vs. plate) is highly sensitive to both composition and heat treatment. Additional transmission electron microscopy work on the Fe-20.2Ni-5.7Mn alloy has shown that four sets of intralath screw dislocation networks are found. All of these dislocations have Burgers vectors of the  $\langle 111 \rangle$  type. Further transmission electron microscopy has been concerned with the dislocations in austenite in the near vicinity of the lath martensite-austenite interface, the present implication being that the austenite dislocation structure on each side of an isolated lath is somewhat different, this possibility is interpretable on the basis of a one-sided thickening or growth mechanism.

*Reports:*

- No. 1-5 in previous editions.  
6. Martensite transformations in Iron Alloys, by C. M. Wayman, Mar 81, 3p. FR: 1 Jan 78 - 31 Dec 80.

**15104 ELECTRODEPOSITION OF CHROMIUM AND COPPER IN MAGNETIC FIELDS**

John Dash  
Portland State University  
SL: BWL SC: ARRADCOM, AMMRC, MERADCOM

A paper describing the effects of coupled electric and magnetic fields on electrolytic processes has

been revised and submitted for publication. This paper includes results with possible technological implications based on a method to either increase or decrease electrolytic cell potential. The idea is that increasing the cell potential may facilitate the deposition of metals such as nickel without co-deposition of hydrogen gas. This could have important implications to the properties of electrodeposited nickel and other metals above hydrogen in the EMF series. At present the idea is unproven. Work on the comparative effect of magnetic fields on protons and on hydroxyl ions in an electrolyte is not yet completed. In the course of structural studies, one electron microscope suddenly exhibited very high dark current and external x-ray emission around the electron gun. With the aid of a helium leak detector, the problem was traced to a poor seal in the gun chamber. Research on the effect of magnetic fields on electrodeposited copper shows that a magnetic field changes both the structure and the properties of these deposits.

#### Reports:

- No. 1-2 in previous editions.
- 3. Reduction Emitted from a TEM Due to an Air Leak, by J. Dash et al, *Bull Electron Microscopy Soc Am*.

### 15916 ELECTROPLATING OF REFRACTORY METALS AND ALLOYS FROM FLUORIDE MELTS

R. A. Bailey  
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SL: RWL SC: FCSCWSL

Electroplating of refractory metals from fused fluorides requires that the reduction process not be simple if nondendritic deposits are to be obtained. Chromium plating from the LiF-NaF-KF eutectic results in smooth deposits at temperatures above 900C, but increasingly more dendritic deposits as the temperature is lowered, although Mellors and Senderoff performed a chronopotentiometric study of Cr(III) reduction in this melt, this was at 750C only. Chronopotentiometry and cyclic voltammetry have been applied to this system over a temperature range of 550C to 1000C, and over a range of Cr(III) concentrations, and the results show extensive differences in the electrochemistry over this temperature range. For these measurements, a 3-electrode system was employed, using a platinum quasi-reference electrode,

platinum working electrodes and a vitreous carbon auxiliary electrode.

### 15979 THE MECHANISM OF FRETTING FATIGUE CRACK INITIATION IN MEDIUM STRENGTH STEELS

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SL: MERADCOM SC: MERADCOM, BWL,  
ARRADCOM ARRADCOM, TARADCOM,  
AVRADCOM

Effects on the fretting fatigue life of quench and tempered (Rc 42) 4130 steel due to moisture content and oxygen partial pressure in the environment have been examined. In addition, fretting pads of various hardnesses and radii of curvature have been used to aid in the development of a mathematical model of the contact mechanics. In general specimens tested in dry air and dry oxygen have longer lives than specimens tested in wet conditions at a given normal stress. In air the fatigue life is about 4 times as long in dry conditions and in oxygen it is about 1.2 times as long, at a normal pressure of 50 MPa. At a normal pressure of 60 MPa the fatigue life is about 1.9 times longer in dry condition for both air and oxygen environments. The effect of oxygen partial pressure is increasingly more pronounced with lower applied normal stresses. At 60 MPa fatigue lives are about the same for wet and dry oxygen as they are for wet and dry air. At 50 MPa fatigue lives are about twice as long in oxygen and at 42.5 MPa they are about 3 times as long. The most important factor in determining the total fatigue life seems to be the relative slip amplitude between the fatigue specimen and the fretting pads. In general it was observed that fatigue life is minimum at about 40  $\mu\text{m}$  of slip amplitude. Experiments were conducted on 4130 steel to study the change in fretting fatigue life between quench and tempered specimens and surface-treated specimens. Carburization was chosen to be the surface hardening treatment and de-carburization to be the surface softening treatment. Two case depths were studied in the content of decarburized specimens to investigate the effect of case depth on fretting fatigue life. Preliminary test results have indicated that carburized specimens have longer fretting fatigue lives than quench and tempered specimens,

whereas decarburized specimens have a shorter life. Also, decarburized specimens with a deeper case showed the least resistance to fretting and had consistently short lives.

**Reports:**

No. 1-2 in previous edition.

## 16062 SILICON CARBIDE ALLOYS

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SC: AMMRC, EUSTIS, LANGLEY, BWL,  
MERADCOM, MICOM, TARADCOM

The high temperature graphite element hot press is now routinely in operation. SiC-AlN Specimens, ranging from 0 w/o AlN, were hot-pressed to high density without boron additions. The hot-pressing temperature varied from 1950 to 2100°C. Microhardness was measured. The data revealed a linear relationship between microhardness and AlN content. X-ray diffraction studies of the hotpressed specimens reflected the lattice parameters changing with composition. Microscopic analysis of the samples revealed a two-phase microstructure. The physical and chemical properties of selected SiC-AlN powders were determined. Young's elastic modulus has been measured on a variety of materials using the electromagnetic method. Microhardness is being routinely measured. A high temperature furnace has been assembled and will be used for annealing studies.

**Reports:**

1. Fabrication and Characterization of SiC-AlN Alloys, by William Rafaniello et al, *J Mater Sci*.

## 16296 SURFACE MELTING AND ALLOYING OF METALS BY CO<sub>2</sub> LASERS

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C. T. Walters  
B. P. Fairand

Battelle Columbus Laboratories

SL: FCSCWSI, BWL SC: CERL, MERADCOM,  
AMMRC, LEWIS

Additional studies were conducted on the vacuum coupling of the TEA laser pulse to polished iron and Fe-C specimens. Open shutter photography of

the specimen surfaces revealed plasma formation at energies below that necessary to form smoothly melted surfaces. A one-dimensional laser heating computer program was developed to model these short interaction time events. The program handled temperature dependent specific heat and laser absorptivity as well as fusion and vaporization effects. Results were compared to measured values of the thermal coupling coefficient versus incident laser energy and to observe plasma threshold energies. TEA laser pulse melting applied to homogenized FeC coupons showed less cratering than for the same treatment applied to as-cast coupons. The homogenizing melt passes eliminated the large carbon precipitates in the casting. The large carbon particles acted as preferential absorbers in the cast material thereby creating craters on the laser melted surface. Homogenizing passes made with the AFML 10KW laser in the top hat mode were able to produce broad melts about 50  $\mu$ m deep on grit-blasted Fe-4C specimens. For the scan speeds and beam sizes employed, this depth is surprisingly small. Additionally, microhardness data showed a fusion zone hardness nearly equal to that of the heat affected zone, both of which were 50 percent higher than the original casting hardness.

## 16348 BASIC RESEARCH IN WELDING FLUXES AND FILLER MATERIALS

David L. Olson  
Colorado School of Mines

SL: AMMRC SC: MERADCOM

Weld metal acicular ferrite is generally correlated with high fracture toughness. The quantity, size, and distribution of AISI 1020 carbon steel weld metal ferrite was measured and related to weld metal and flux composition. The weld metal oxygen content has been correlated with a variety of reagent grade manganese silicate submerged arc welding flux compositions. The amount of acicular ferrite is definitely related to the weld metal oxygen content, although some flux additions show a better correlation than others. Efforts to understand the role of weld metal oxygen on the microstructure are in progress. Optical and electron microscopy is being used to characterize the inclusion type and/or size distribution and to determine if inclusions are the nucleation sites for



upper transformation products during continuous cooling. The MnO-SiO-Li<sub>2</sub>O submerged arc welding flux system has demonstrated the achievement of typical "low oxygen type" acicular ferrite at high oxygen contents. Efforts are being made to understand the specific behavior of this welding flux on the microstructure. Investigations to determine the influence of weld metal oxygen contents on weld penetration and arc instability have been completed. The concepts of Patchett and Dancy were tested and no general evidence was found to support their arguments. Penetration was found to correlate well with weld metal oxygen contents. For some flux systems definite increases in penetration with an increased oxygen was determined while for other flux systems just the opposite was found.

#### Reports:

No. 1-5 in previous editions.

6. The Influence of Ionic Character on Welding Flux Behavior, by T. L. Reinhart et al, *Proc of Intl Conf on Welding Research in 80's*.

### 16782 PULSED ELECTRODEPOSITION OF AMORPHOUS AND COMPOSITE ALLOYS

R. Weil

Stevens Institute of Technology

SL: ETDI, BWL SC: MERADCOM, AMMRC

The objective of the research is to investigate the effect of pulse plating on the compositional variation in electrodeposited alloys. There are two tasks. For the first task, the effect of pulse plating on producing a more uniform composition and reducing hydrogen codeposition is being studied. For the second task, pulse plating is being investigated to enhance compositional variations so as to enable the creation of composite materials by subsequent heat treatment. Amorphous nickel phosphorus alloys are being studied. In order to avoid using proprietary plating solutions, the properties of Ni-P alloys from electrolytes of various compositions were determined. The phosphorus content of the alloys varied from 9.4 to 16.5% depending on how much metal has been plated previously. This variation can be controlled by maintenance of the hypophosphite content. The mechanical properties were determined by a bulge test. The true strain at fracture decreased from 6.3

to 1.3% and the true fracture stress increased from 53 to 58 megapascals with increasing %P. Methods are being developed for thinning the cross section for transmission electron microscopy to determine local compositional variation with thickness. The pulse plating duty cycles will be based on these results. Nickel-molybdenum alloys are being studied for the second task. The effects of Ni:Mo ion ratios and the current density on the structure are being investigated. The best deposits were obtained with a 10:1 ratio. The principal ingredients in the plating solutions are nickel sulfamate and sodium molybdenate.

### 16824 FORMATION OF MONOLITHIC OXIDE MATERIALS BY CHEMICAL POLYMERIZATION

B. E. Yoldas

Westinghouse R&D Center

SL: AMMRC SC: ETDI, MICOM

Investigation of the structure of glasses made from polymerized gels is proceeding through the utilization of infrared spectroscopic techniques. Studies have indicated that the composition and structure of these glasses are affected significantly by the amount of water used in the initial hydrolysis of the starting organometallic compound. In the titania and silica systems, for example, gels can be formed such that they will contain from 65 to over 90% equivalent oxide by weight when calcined at temperatures as low as 500C. The non-oxide portion of these gels consists basically of hydroxyl groups, with small amounts of alkyl groups also present. Interestingly, the hydroxyl content of gels varies inversely with amount of water used for hydrolysis; that is, gels with more hydroxyl groups are produced by using less water in hydrolysis. Once the gel is formed, it must be pyrolyzed to the final oxide. Studies of this heating process indicate that non-oxide components are liberated at two distinctly different rates: a gradual continuous liberation takes place between 150 and 500C, during which about 5 weight percent is lost, and a second, more vigorous liberation occurs in the range of 200-250C, which may be responsible for as much as a 40% weight loss. This latter reaction may be responsible for the breaking of the gel network, disrupting monolithicity. Because of its importance in the practical application of the whole process, this point will be investigated in greater detail.

# 16916 ANALYSIS OF MICROSTRUCTURE AND STRENGTHENING MECHANISMS IN LASER SURFACE ALLOYED Fe-Cr and Fe-C-Cr ALLOY SYSTEMS

William E. Wood  
Oregon Graduate Center  
SL: FCSCWSL SC: AMMRC

The program to date is studying fundamental aspects of laser surface alloying of chromium on commercial pure iron to produce different surface alloy compositions and microstructures. Three main tasks are being emphasized. They include: (1) The influence of laser beam scan rate. (2) The effects of chromium coating thickness. (3) The effects of internal gas associated with the coating on melt zone geometry, alloy compositions, solidification aspects and microstructural features. It appears that small variations in output power, of the order of 50 to 100 watts and in spot size of the order of 0.005", cause a significant difference in melt penetration and width. Studies are currently underway to optimize laser beam parameters and also to study the impact of different shielding gases and various electroplated surface conditions on the laser surface alloying performance. In addition to laser surface alloying of chromium, alloying of chromium and nickel, codeposited chromium and iron, and black chromium electrodeposits are also in progress.

# 17161 THE EFFECTS OF POWDER METALLURGICAL PROCESSING AND INTERMEDIATE THERMAL MECHANICAL TREATMENT ON THE FATIGUE PROPERTIES OF HIGH STRENGTH ALUMINUM ALLOYS

S. B. Chakraborty  
E. A. Starke, Jr.  
Georgia Institute of Technology  
SL: FCSCWSL SC: LEWIS, MICOM, AMMRC, MERADCOM

Microstructure and texture of materials may be controlled to a large degree to obtain an improvement in a given mechanical property. This is usually achieved at the expense of another property. For example, one may get an improvement in fatigue stress-life or strain-life

behavior by reducing the slip length through microstructural control. However, usually this also leads to a decrease in the resistance to fatigue crack growth. This research is directed towards understanding fatigue mechanisms in terms of microstructural variations produced by processing (i. e., ITMT and rapid solidification) and in optimizing microstructure to enhance fatigue initiation and crack growth resistance. Four CT-91 alloys with different microstructures and textures were acquired for comparison of fatigue properties. Ingot alloys were conventionally processed (IMCP) or ITMT processed (IMIP). Powder alloys were conventionally processed (PMCP) or ITMT processed (PMIP-AR) and aged to the T7E70 condition. The microstructural analysis of IMCP showed a 30% degree of recrystallization, with large elongated pancake-shaped grains and a sharp texture. PMIP-AR was shown to have a 22% degree of recrystallization, with small grains and a weak texture. ITMT processing produces a more homogeneous microstructure and gives more uniform mechanical properties, especially for powder alloys. The powder ITMT processed alloy shows the best combination of fatigue crack initiation and propagation resistances.

# 17377 MICROWAVE SINTERING OF CERAMICS

D. Lynn Johnson  
Morris E. Brodwin  
Northwestern University  
SL: ETDL, MICOM SC: CENCOMS, MERADCOM

The objective of this research is to evaluate the novel approach of microwave sintering of ceramic powder compacts, and to investigate the effect of this rapid heating technique on sintering kinetics. The program will include the following: (1) Develop an automated microwave sintering apparatus with absorbed power monitoring capability, variable sample feed rate drive, automatic temperature monitoring and recording, and automatic control of resonant frequency and coupling. (2) Assess the usefulness of microwave sintering for a variety of ceramic materials. (3) Perform a detailed study of the sintering and microstructure in  $\beta''$ -alumina including densification rates, grain growth and the relationship between sintering parameters.

microstructure, electrical and mechanical properties. (4) Perform a detailed study of a suitable solid state sintering system (as determined in (2) above), particularly, the mechanism(s) of rapid densification over a wide range of particle sizes.

#### 17439 PREPARATION AND PROPERTIES OF LOW TEMPERATURE DEPOSITED SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> FILMS

S. R. Butler  
Lehigh University  
SL: HDL SC: ETDL, NVEOL

The objective of the research is to study the chemical and structural origins of charge trapping centers in thin oxide dielectric films prepared by low temperature growth and deposition processes. The silicon oxide and aluminum oxide films will be prepared by low pressure chemical vapor deposition over 50 Å thermally grown SiO<sub>2</sub> and Si. Chemical analyses will be performed by Rutherford backscattering and sputter induced photon spectroscopy with hydrogen detection capability. The scanning transmission electron microscope will be used for structural characterization. The electron injection and trapping behavior of these structures will be investigated by avalanche injection, internal photoemission, photo I-V and thermal and photodepopulation measurements.

#### 17455 SOLIDIFICATION OF HIGHLY UNDERCOOLED LIQUID DROPLETS

J. H. Perepezko  
University of Wisconsin  
SL: FCSCWSL, BWL SC: MERADCOM, AMMRC

The objective of this research is the investigation of phase selection kinetics and microstructural evolution during solidification of non-equilibrium metastable metals of highly undercooled and hyper-cooled melt. This research will include: (1) Utilization of the liquid emulsion droplet techniques to examine metastable phase reaction kinetics by differential thermal analysis (DTA). (2) Investigation of the effect of pressure on the formation and stabilization of metastable phases.

(3) Development of the droplet-convulsion technique to include experimental methods for investigations above 500°C, the current limit. This will allow studies of non-equilibrium phases of aluminum and other metals and alloys of intermediate range melting point. (4) Studies of the formation of amorphous phases and their subsequent compaction into bulk shapes. Several features of the microstructural modification associated with the rapid solidification that follows the onset of nucleation in a highly undercooled liquid sample have been examined during the initial period of the project. The experimental approach that is being applied involves the use of the droplet technique to generate liquid samples which exhibit a deep undercooling in the range of 0.3 to 0.4 T<sub>M</sub> before solidification. The amount of undercooling can be controlled at different levels by the use of a variety of droplet surface coatings with different catalytic potencies so that microstructural development may be studied as a function of undercooling. The capability to produce droplet samples with a variety of nucleation temperatures has been developed for Bi-Sb alloys which form a continuous series of solid solutions.

#### Reports:

1. Metastable Phase Formation in Undercooled Liquids, by J. H. Perepezko et al. *Synthesis and Properties of Metastable Phases*, ed by E. S. Machlin et al, p31 1980. AD A097 910

#### 17536 PLASMA-ENHANCED DEPOSITION AND PROCESSING OF METAL SILICIDES

D. W. Hess  
University of California at Berkeley  
SL: ETDL, HDL

The objective of the investigation is to provide basic understanding of the relationship between plasma parameters and materials properties of silicide films. The program will include: (1) Determination of the effects of processing parameters (e. g. rf plasma power, substrate temperature, source vapor composition and pressure) on the nature of the disilicide thin films (e. g. residual stress, electrical resistivity, structure). (2) Characterization of the deposited films.

# 17643 STUDIES OF METASTABLE MICROCRYSTALLINE AND GLASSY ALLOYS: INTER AND INTRADISCIPLINARY RESEARCH

N. J. Grant

Massachusetts Institute of Technology

SL: FCSCWSL, AMMRC SC: AMMRC, MERADCOM, ETDL

The objectives of this research are to investigate metastable microcrystalline and amorphous alloys in a multifaceted, multi-investigator program which will include alloying studies of microcrystalline aluminum-base, nickel-base, Ni-Mo-base, and Fe-Gr-Al-base alloys; studies of stabilization, crystallization and properties of glassy alloys based on Fe-metal-metalloid systems; and studies of hydrogen assisted comminution and consolidation of metastable alloys. The research effort will be divided between MIT and Northeastern University. The MIT effort will focus on the first two general areas in the objective using ultrasonic gas atomization (USGA), roll quenching (RQ), combinations of these two techniques, and USGA plus a flat spinning disc to prepare alloys of these compositions. Consolidation and mechanical property measurements in addition to microstructural characterization will be the main emphasis in the first group of alloys. In the second group of alloys, corrosion and oxidation resistance and magnetic properties of selected compositions, in addition to consolidation and mechanical properties, will be studied. The Northeastern group will use the approach of melt spinning to prepare a wide range of alloy compositions including metallic glasses, metastable solid solutions, and microcrystalline, near-equilibrium alloys of metallic elements, steels, and others. The comminution of these ribbons and flakes to fine powders will be studied using hydrogen embrittlement. Dehydriding, consolidation of the powders and property studies at MIT which parallel those listed above will be performed.

# 17877 MICROSTRUCTURAL CONTROL AND PROCESSING OF LIQUID PHASE SINTERED ALLOYS

T. H. Courtney

J. K. Lee

Michigan Technological University

SL: AMMRC, BRL SC: FCSCWSL, MERADCOM, BWL

The objective of this research is to study the control of microstructure in liquid phase sintered alloys

## D. Effects of Structure, Defects, and Composition

and like alloys which undergo subsequent deformation in order to produce powder metallurgical composites of combined high strength and toughness. This investigation will include: (1) Microstructural studies of low solid phase contiguity alloys of Fe-(Cu-Mn) and Mo(Fe-Ni) during liquid phase sintering, and (2) the effect of deformation processing on microstructure and properties of these liquid phase sintered alloys.

## D. EFFECTS OF STRUCTURE, DEFECTS, AND COMPOSITION

# 14638 EQUATION OF STATE OF SIMPLE METALS

A. L. Ruoff

N. W. Ashcroft

Cornell University

SL: BWL SC: AMMRC, MICOM

Isothermal measurements of the equation of state of a large potassium crystal, which was determined to 7 kbar produced exciting results and these will soon be published. Subsequent to these measurements the fabrication of a new high pressure vessel built from a non-magnetic titanium alloy has been partially completed. This has been designed to extend the equation of state data to much higher pressure. A new pressure sensor using a temperature-compensated Zener diode has been developed. It has been designed to measure pressures up to 30 kbar. Repeated tests of the sensor have already been carried out at 10 kbar; one pressure run has been conducted at 16 kbar. The results achieved have been extremely promising in the sense that the device has superior performance compared with others of its kind. A new way of approaching the problem of van der Waals interactions in metals was developed: it involves the idea of screened polarization waves. The calculations of the equation of state of potassium at high pressure, which were completed a year back, were modified to include this new development. Calculations on the non-equilibrium properties of metals are also being made.

## Reports:

No. 1-2 in previous editions.

3. Isothermal Equations of State of Potassium, by Kwang Yul Kim et al, *J Appl Phys*.



# 15124 MOLECULAR DESIGN AND SYNTHESIS OF NEW NONCRYSTALLINE SOLIDS

R. Roy

Pennsylvania State University

SL: AMMRC SC: ETDL, ARRADCOM, HDL,  
MERADCOM, MICOM

Due to the high energy quenching rate of vapor-to-solid transformation processes, such as rf-sputtering which is used extensively in this study, noncrystalline or highly metastable thin films can be prepared over a much wider composition range than conventional glasses. This leads to low surface and bulk mobility of the depositing atoms which results in various highly metastable structures. Work has progressed or has been completed on several aspects of the nature of the metastability. First, it has been shown that the amount of atomic clustering is directly related to the disorder in the material. Both the percentage of clusters and their spatial distribution has been investigated using a high-resolution atom probe field ion microscope. Second, for composite materials in which a metal is finely dispersed in an insulating matrix, it was shown that over a wide range of conditions and compositions, the metal phase is microcrystalline but with a size below that detectable by x-ray diffraction. Such fine crystallites (10-80Å) have been prepared and characterized in three different materials. Third, for completely noncrystalline thin films it was found that the morphology is distinctly different than that of a glass (liquid-to-solid transformation). The model of the morphology of noncrystalline thin films is the composite result of all the various results described above plus ongoing work on the preparation and characterization of "a-WO<sub>3</sub>" film.

## Reports:

- No. 1 in previous edition.
- Low Frequency Modes in Amorphous Boron-Rich Alloys, by Jeffrey S. Lannin et al, *Phys Rev Let*.
- Field Evaporation of Cluster Ions from Amorphous and Polycrystalline Thin Films, by S. V. Krishnaswamy et al, *Appl Phys Let*.
- Ba:TiGe<sub>2</sub>O<sub>8</sub> and Ba:TiSi<sub>2</sub>O<sub>8</sub> Pyroelectric Glass-Ceramics, by A. Halliyal et al, *J Mater Sci*.
- Pyroelectric Glass-Ceramics, by G. J. Gardopee et al, *Appl Phys Let* 36, 817 (1980).
- Cluster Formation in Amorphous and Polycrystalline Thin Films by S. V. Krishnaswamy et al, *Thin Sol Films*.
- Fine Structure of Gold Particles in Thin Films Prepared by Metal-Insulator Co-Sputtering, by R. A. Roy et al, *Thin Sol Films*.

- Microdiffraction from Gold Microcrystals, by J. M. Cowley et al, *Scanning Electron Microscopy*.

# 15433 SYNTHESIS OF NEW SOLID ELECTROLYTES

Ephraim Banks

Polytechnic Institute of New York

SL: ETDL, AMMRC SC: MERADCOM

Work has been completed on ZrP<sub>2</sub>O<sub>7</sub> solid solutions. The evidence is that sodium and lithium ions are mobile in this structure, while potassium and silver ions are considerably less mobile. The best conductivity, for Li<sup>+</sup>, was only  $1.5 \times 10^{-4}$  (ohm-cm)<sup>-1</sup> at 300C, in Zr<sub>0.65</sub>Li<sub>0.35</sub>P<sub>2</sub>O<sub>7</sub>. Samples of (Li-Y) substituted materials had higher conductivity than Li-In samples of the same Li concentration. This is ascribed to enlargement of the "bottlenecks" between adjacent cation sites, caused by expansion of the lattice. Unfortunately, the amount of yttrium substitution is limited to about x = 0.20. The silicon-substituted materials, e.g., Zr(Li, Si) x P<sub>2</sub>-xO<sub>7</sub>, showed a lattice expansion with increasing Li, Si concentration. These samples initially showed the expected high conductivities; however their conductivity rapidly decreased with time, apparently the result of an electrode reaction. A layer of a white powder was formed, which had an x-ray pattern resembling that of LiH<sub>2</sub>PO<sub>4</sub>.

## Reports:

- Conductivity of Reduced Solid Solutions in the System Na<sub>2</sub>O-Al<sub>2</sub>O<sub>3</sub>-TiO<sub>2</sub>, by Y. Avigal et al, *Mater Res Bull* 15, 1381 (1980). AD A095 686

# 15771 MECHANISM OF HYDRATION OF CALCIUM SILICATE-BASED HYDRAULIC BINDERS

J. P. Skalny

I. Jawed

Martin Marietta Laboratories

SC: AMMRC, WES, CERL

Surface hydration studies of β-C<sub>2</sub>S using the techniques of ESCA and high resolution SEM indicate a hydration mechanism similar to C<sub>3</sub>S although it hydrates at a slower rate compared to

C<sub>3</sub>S. AEM studies on hydrated C<sub>3</sub>S and cement pastes (cured up to 30 years) show significant differences in Ca/Si ratios in C-S-H. C<sub>3</sub>S and  $\beta$ -C<sub>3</sub>S hydrate by a similar mechanism; however, the rate of C<sub>3</sub>S hydration is slower. Presence of gypsum modifies the rate of products of C<sub>3</sub>S hydration. AEM studies revealed the compositional variations of C<sub>3</sub>S and cement hydration products as a function of time (degree of hydration).

#### Reports:

- No. 1-11 in previous edition.
12. Hydration of Calcium Silicates, by Jan Skalný, *Cement-WAPNO-GIPS* 33, 286 (1980). (in Polish) AD A099 547
  13. Hydration of Tricalcium Silicate - A Study of High Resolution Scanning Electron Microscopy, by D. Menetrier et al, *Silicates Industriels* 12, 243 (1980). AD A095 840
  14. Effect of Gypsum on C<sub>3</sub>S Hydration, by D. Menetrier et al, *Cement and Concrete Res* 10, 697 (1980).
  15. Cement Hydration: An Overview, by Jan Skalný, pres Mater Res Soc Meeting.
  16. Hydration Studies on Cement and Calcium Silicates, by I. Jawed et al, *World Cement Tech* 12, 124 (1980). AD A099 551
  17. Hydration of Cement, by Jan P. Skalný, *Revista IMCYC* 19, 35, (1981). (in Spanish) AD A099 550
  18. Surface Phenomena During Tricalcium Silicate Hydration, by Inam Jawed et al, *J Colloid and Interface Sci.*

### 15804 MECHANISM OF INSERTION IN HIGH CONDUCTIVITY GRAPHITE INTERCALATION COMPOUNDS

F. Lincoln Vogel  
William C. Forsman  
University of Pennsylvania  
SL: AMMRC, MERADCOM SC: ETDL, BWL

Since establishing the chemical analysis procedure, efforts have continued in investigating the chemical composition and synthesis of the graphite-FeCl<sub>3</sub> compounds. In order to reduce impurities, FeCl<sub>3</sub> was prepared *in situ* for intercalation. Iron wire was placed in a glass tube attached to a vacuum line. Chlorine was passed through a P<sub>2</sub>O<sub>5</sub> drying tube, condensed in a dry-ice acetone trap, and distilled into the reaction tube. FeCl<sub>3</sub> was readily formed by gently heating the iron wire. The solid was resublimed several times in the presence of chlorine to convert traces of ferrous chloride into ferric chloride. Excess chlorine and ferric chloride were then distilled into the side tube where the graphite crystal was placed and sealed off. Chemical analysis of these resulting G-FeCl<sub>3</sub> compounds found that

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the Cl/Fe mole ratio averaged around 2.4. Weight uptake and x-ray pattern indicated a first stage compound. Electrical conductivities of these compounds were comparable to those that were prepared by commercial FeCl<sub>3</sub>, maybe slightly higher by this method. Chemical analysis favors the argument that the iron atom has been partially reduced. However, the exact chemical state of the intercalants remains to be determined.

#### Reports:

- No. 1-2 in previous edition.
3. Mechanism for Intercalation of Graphite by Antimony Pentafluoride, by William C. Forsman et al, *Chem Commun.*
  4. Mobility of Spacer Molecules in Graphite Nitrate, by W. C. Forsman et al, *Mater Sci and Eng.*

### 15824 EFFECT OF ATOMIC AND ELECTRONIC STRUCTURE ON CARRIER TRANSPORT IN THIN SILICON OXIDE AND NITRIDE MATERIALS

Martin Peckerar  
Westinghouse Electric Corporation  
SL: ETDL, HDL SC: NVEOL

Studies of process-induced variability of injection barriers in thin oxide films have been concluded. UV photoemission spectra indicated the position of the oxide valence band edge with respect to the vacuum level was constant (to ~0.5V) regardless of the process used to form the oxide. Air grown, boiling nitric acid grown and 750K dry grown oxide were studied. The valence band edge position was essentially constant as a function of oxide thickness for thicknesses ranging from ~10Å to 340Å. A similar study of the conduction band edge of these oxides using UV absorption measurements indicated similar consistency of this band edge with respect to the vacuum level. The MNOS memory nitride (LPCVD) optimization experiments have produced some definitive data. Memory structures were fabricated by nitride deposition under varying conditions: temperature (700 and 750C), pressure (150 to 600 mtorr), dichlorosilane/ammonia reactant gas ratio (1:3 to 1:14.4), prepurge time (0.5 to 15 mins) and post deposition hydrogen anneals at 900C. The characteristics derived as a result of these parameter changes were C-V pulse response (memory clear window size, write speed and

trapping), current-voltage measurements (relative leakage), memory retention and dielectric breakdown. Correlating all the data on the prepurge time variation shows significant effect on memory parameters except that the shortest prepurge time (0.5 mins) had about 1 volt less  $-V_{\pi}$ . At a reduced deposition pressure of 150 mtorr, the C-V pulse data indicated (by a shift of the curves in the positive direction) that there was less fixed positive charge in the nitride. The memory window size, however, remained unchanged.

#### 15999 ROLE OF MICROSTRUCTURE IN EPITAXIAL REGROWTH IN ALUMINUM-SILICON INTERFACES

R. A. Armistead  
T. J. Magee  
Advanced Research and Applications Corp.  
SL: ETDL SC: HEL, NVEOL

Solid phase regrowth of Si on sapphire has been investigated in the Si(amorphous)/Al(poly)Al<sub>2</sub>O<sub>3</sub> (crystal) system. Using transmission electron microscopy, scanning electron microscopy, Auger electron spectroscopy and Hall effect measurements, it has been shown that Si is transported through an Al film at 550 degrees C to produce p-type Si films on the sapphire substrate. The growth process has been shown to be initiated at Si nucleation sites on the substrate. These sites expand by mass accretion forming island structures that coalesce to yield continuous large grained polycrystalline Si films on the sapphire surface. In addition, experiments are being conducted on as-deposited (CVD) and scanning-laser-annealed Si films on sapphire to assess the mechanisms of laser-induced recrystallization and perfection of regrown layers. Experiments were also conducted in conjunction with J. F. Gibbons (Stanford University) in support of another ARO-funded program on CW-laser-assisted diffusion and activation of Sn in GaAs from a SnO<sub>2</sub>/SiO<sub>2</sub> source. These experiments have a direct relationship to the study of metal-semiconductor reactions and provide an opportunity to investigate another metal-semiconductor system.

##### Reports:

1. Seeded and Limited Regrowth of Si Over SiO<sub>2</sub> by CW Laser Annealing, by T. J. Magee et al, *Appl Phys Lett* 38, 248 (1981). AD A097 701

2. Scanning CW Laser Annealing and Bridging Epitaxy: Seeded and Limited Seeding Regrowth of Si Over SiO<sub>2</sub>, by T. J. Magee et al.
3. Solid Phase Regrowth of Si on Sapphire in the Si/Al/Al<sub>2</sub>O<sub>3</sub> System, by T. J. Magee et al, *J Appl Phys* 51, 3973 (1980).
4. CW Laser Assisted Diffusion and Activation of Tin in GaAs from a SnO<sub>2</sub>/SiO<sub>2</sub> Source, by Y. I. Nissim et al, *J Appl Phys*.

#### 16058 PHASE DIAGRAMS OF III-V COMPOUNDS BY THE STEP GRADING TECHNIQUE

J. R. Hauser  
North Carolina State University  
SL: ETDL, ERADCOM SC: NVEOL, AMMRC

InGaP layers which exhibit the phenomenon of lattice pulling have been successfully grown on GaAs substrates. Electron microprobe analysis of "latticed-pulled" layers indicates that less than 1% As is present. This suggests that As diffusion is not responsible for lattice pulling. Solidus and liquidus data for InGaP on GaAs matches well with data for InGaP on InGaAs. Simple solution fitting of InGaP solidus data has been successful. However, liquidus fitting, while better than previous predictions, is only marginally adequate. Assembly of a sorbtion pump system is nearing completion. A paper describing the results of research into InGaP is being prepared.

##### Reports:

No. 1-2 in previous edition.

#### 16089 THERMODYNAMICS AND KINETICS OF INTERSTITIAL SOLID SOLUTIONS

R. B. McLellan  
Rice University  
SL: AMMRC SC: MERADCOM

The following experimental projects have been completed: (1) Measurements of the diffusivity and permeability of hydrogen in vanadium. (2) Measurements of the diffusivity of hydrogen at low temperatures (~300 - 400K) using a new electrochemical technique. (3) Measurements of the diffusivity of hydrogen in a series of nickel-based alloys containing between 1 and 14 at. % vanadium. The diffusivity of H as a function of temperature has been measured using the permeability time lag

method in a series of Ni-V solid solutions. The V-composition range 0-20 at. %V was spanned. It was found that the H-diffusivity is remarkably insensitive to the V-concentration even though there is no doubt that V-atoms create trapping sites in the Ni lattice. The constancy of the diffusivity is explained by the lattice dilations accompanying the insertion of V-atoms into the lattice enhancing the jump frequency between sites not neighboring V-atoms. It has been shown during this period that the thermodynamic behavior of H-atoms in binary alloys at low temperatures is properly described by Fermi-Dirac statistics. Normally the behavior resulting from overfilling certain energy levels cannot be experimentally observed because at high-temperatures the occupation-density of the energy levels is low and the H-atoms act as Boltzons. However, the mobility of H in Pd and the Group V metals is so large that both thermodynamic and kinetic measurements are possible at temperatures low enough to observe Fermi-Dirac effects.

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2. The Thermodynamics of Palladium-Based Solid Solutions Containing a Noble-Metal Substitutional Solute and Hydrogen, by Rex B. McLellan et al, *J Phys Chem Sol* 42, 157 (1981).
3. Diffusivity of Hydrogen in Nickel-Vanadium Solid Solutions, by W. M. Yei et al, *Acta Met* 28, 1437 (1980). AD A095 243
4. Electrochemical Methods for Measuring Diffusivities of Hydrogen in Palladium and Palladium Alloys, by Reiner Kirchheim et al, *J Electrochem Soc* 127, 2419 (1980). AD A095 262
5. The Effect of Substitutional Impurity Atoms on the Diffusivity of an Interstitial Species, by Rex B. McLellan et al, *J Phys Chem Sol* 41, 1241 (1980). AD A095 246
6. Thermodynamics of Dilute Platinum-Hydrogen Solid Solutions, by Wei-Ming Yei et al, *J Less-Common Metals* 64, 11 (1979).
7. Trapping of Hydrogen in Iron, by Rex B. McLellan, *Scripta Met* 14, 513 (1980).
8. Diffusivity Permeability and Solubility of Hydrogen in Platinum, by Hiroji Katsuta et al, *J Phys Chem Sol* 40, 697 (1979).
9. Solid Solutions of Pd Containing Hydrogen and a Noble-Metal Substitutional Component. Part I: Thermodynamic Behavior, by M. Yoshihara et al, *Acta Met*.
10. Solid Solutions of Pd Containing Hydrogen and a Noble-Metal Substitutional Component, Part II: Kinetic Behavior, by M. Yoshihara et al, *Acta Met*.
11. Thermodynamics of Body-Centered Cubic Iron-Molybdenum-Nitrogen Solid Solutions, by M. Yoshihara et al, *Acta Met* 29, 471 (1981).
12. Diffusivity and Permeability of Hydrogen in Vanadium, by Hiroji Katsuta et al, *JIMIS-2 Int Conf on Hydrogen in Metals*, p113, 1980.

#### D. Effects of Structure, Defects, and Composition

13. The Diffusivity of Hydrogen in Deformed Palladium, by Rex B. McLellan, *Scripta Met* 15, 501 (1981).

#### 16355 ROLE OF COUPLING AGENTS ON METAL-POLYMER ADHESION

N. H. Sung

S. P. Sung

Massachusetts Institute of Technology

SL: AMMRC SC: CSL, HDL, MERADCOM, NLABS, LANGLEY

The structure of organo silane or organo titanate adsorbed on single crystalline aluminum oxide was studied by a combined technique of Fourier transform IR spectroscopy and electron spectroscopy for chemical analysis. Organosilanes, specifically amino-propyl or vinyl silanes, appeared to have undergone hydrolysis and polymerization to form polysiloxane network, where a substantial amount of silanols or dimers/trimers are also present. The exact composition of adsorbed silane seems to depend on the reaction conditions such as concentration of silane in aqueous solution, pH or drying conditions. For example, extensive drying tends to increase cross-linking, as indicated by solubility characteristics and IR spectra. In contrast, organo titanate, namely monoisopropoxy tristearoyl titanate, shows little change in its IR spectra when adsorbed. Only when the adsorbed titanate is heated above 70C for an extended period, do some spectral changes occur, suggesting the disappearance of an ester of stearic acid (possibly isopropoxy stearate). It is known that monoalkoxy tricarboxylate titanates are unstable above 10C, leading to decomposition products such as the ester mentioned above. Other than the fact that adsorbed organo titanate may contain such decomposition products, it does not appear that adsorbed titanate is polymeric in nature. In order to correlate the structure of adsorbed organo silanes or organo titanates with their joint strength, the peel strength of a sapphire-polyethylene joint was measured as a function of concentration and drying conditions. In the absence of a coupling agent, 180C peel strength was very low. A sharp increase in peel strength was seen as the concentration of amino silane was increased from 0.05 to 2% with a maximum strength (2 kg/cm) obtained at 2% concentration. Beyond 2%, peel strength levels off. Peel strength with adsorbed titanate is much lower as compared with adsorbed silane, but it increases



significantly after removing the stearic acid ester from the titanate. Still, peel strength is much lower than with silane. Since chemical reaction between silane and polyethylene is unlikely, the interaction responsible for the improved strength must, therefore, be physical in nature.

#### Reports:

- No. 1 in previous edition.
2. Role of Organo Silanes and Organo Titanates in Promotion of Adhesion Strength of Aluminum Oxide-Polyethylene Joint, by N. H. Sung et al, *Proc 36th Annu Tech Conf.*

### 16423 STRUCTURE AND PROPERTIES OF INTERCALATED GRAPHITE FIBER-POLYMER COMPOSITES

W. C. Forsman

University of Pennsylvania

SL: MERADCOM, AMMRC SC: ETDL

Studies have indicated that graphite can be intercalated at 300-350°C by the gas-phase complex that formed between  $\text{AlCl}_3$  and  $\text{MnCl}_2$ . However, chemical analysis of the products indicates that only  $\text{AlCl}_3$  is inserted into the lattice until intercalation reaches stage-2. Then  $\text{MnCl}_2$  is inserted on further intercalation to stage-1. Work is in progress to rationalize that behavior with the current understanding of the chemistry of intercalation. In order to continue experiments with present facilities, the technique for impregnating graphite fibers was scaled down. In addition, a new epoxy resin system was adopted using a polyamide curing agent. Compared to amines, polyamides are relatively non-toxic. Experimental evidence indicated that the modified procedure, as well as the resin system, was adequate for initial studies. Composites of GY-70 fiber, intercalated to about stage-4 with nitric acid, were made. Preparation of specimens of these composites for short-beam shear tests is in progress. Composites of HMU fibers intercalated with nitric acid are presently being prepared. Short beam shear tests will be made to determine which intercalated fiber results in composites with optimum shear strength.

### 16438 MECHANISMS AND KINETICS OF RESIDUAL STRESS RELAXATION IN CERAMICS

D. P. H. Hasselman

Virginia Polytechnic Institute and State University

SC: AMMRC, MERADCOM, TARADCOM, MICOM, LEWIS

The objective of the present research program is to develop an experimental as well as theoretical understanding of all possible mechanisms which control the competitive processes of residual stress relaxation and fatigue. An analysis was conducted of elastic creep by crack growth in polycrystalline oxides. For this purpose, an extensive computer program was compiled which permitted calculation of the stress and temperature dependence of creep rates by all possible mechanisms, including crack growth, dislocation motion, Nabarro-Herring and Coble creep. Creep rates were predicted on the basis of mechanistic theories as well as experimental data for the crack growth behavior. Coupling between diffusional and plastic flow phenomena was assumed absent. The information obtained was expressed in terms of deformation maps which indicate the dominant mechanism of creep at a given stress and temperature. The results indicate that creep by crack growth in aluminum oxide occurs by surface or grain boundary diffusion at stress levels immediately below the fracture stress. Also, creep by crack growth is more likely to occur in coarse grained than in fine grained microstructures, in contrast to Nabarro-Herring and Coble creep which shows an inverse dependence between creep rate and grain size. Of significance is the finding that appreciable rates of creep by crack growth can occur at temperatures at which other mechanisms of creep make no measurable contribution to the total creep rate. A further theoretical study underway at this time concentrates on analyzing the principles of "elastic creep", i.e., the time-dependent creep strain which results from a time-dependent change in elastic properties.

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1. Elastic Creep of Brittle Ceramics with Special Reference to Creep by Crack Growth in Aluminum Oxide, by D. P. H. Hasselman et al, *pres Conf on Surfaces and Interfaces in Ceramic and Ceramic-Metal Systems*.
2. "Elastic Creep" of Stressed Solids Due to Time-Dependent Changes in Elastic Properties, by A. Venkateswaran et al, *J Mater Sci*.

3. Thermal Expansion Anomaly Due to Residual Stress Relaxation in a Polycrystalline Aluminum Oxide, by Y. Tree et al, *Proc 8th Intl Thermal Expansion Symp.*
4. Elastic Creep by Crack Growth During Residual Stress Relaxation in a Polycrystalline Aluminum Oxide, by D. P. H. Hasselman et al, *Proc 3rd Intl Symp on the Fracture Mech of Ceramics.*

#### 16448 BASIC RESEARCH ON OXYNITRIDE GLASSES

R. E. Loehman

SRI International

SL: AMMRC, MICOM SC: AMMRC, MERADCOM

The substitution of nitrogen for oxygen in silicate glasses is gaining recognition as a fairly widespread phenomenon. Oxynitride glass formation depends on the close similarities in the local structures of  $\text{Si}_3\text{N}_4$  and the silicates, and is the basis for the observation that oxynitride glasses can be made from silicate systems in which  $\text{Si}_3\text{N}_4$  is appreciably soluble below about 1700C. Typical glass-forming systems are M-Si-O-N and M-Al-Si-O-N, where M is Ca, La, Li, Mg, or Y. Glasses containing as much as 10 at% N have been prepared from these compositions. Extensive properties measurements show that the glass transition temperature, hardness and relative fracture toughness increase, and the thermal expansion coefficient decreases with increasing nitrogen content. This is consistent with a structural model in which trivalent nitrogen substitutes for bivalent oxygen to produce a more tightly linked glass network. This suggests oxynitride glasses may have important technical applications.

##### Reports:

1. Oxynitride Glasses, by Ronald E. Loehman, *Proc of Conf on Frontiers of Glass Sci.*

#### 16740 LOCAL DEFECT ARRAYS AND PROPERTIES OF OXIDES

J. B. Cohen

M. E. Fine

T. Mason

Northwestern University

SC: ETDL

The magnetic properties of W doped  $\text{VO}_2$  have been investigated using both the low field (0-14 kGauss

#### D. Effects of Structure, Defects, and Composition

and 3.5-300K) and the high field (0-90 kGauss and 1.5-90K) Faraday balances in Argonne National Laboratory. The thermoelectric coefficient of cobaltous oxide was measured as a function of temperature and oxygen activity between 1000 and 1500C. Results conform to ideal point defect theory at high temperature and high oxygen activity. Minority electrons are detected near the low oxygen activity phase boundary at every temperature. At low temperature and high oxygen, exponents of the hole concentration exceed that predicted by ideal point defect theory. The atmosphere control system for separate thermopower and high temperature x-ray camera studies has been constructed and tested. Preliminary measurements of the low temperature thermoelectric coefficient support the small polaron model of conduction. Low temperature measurements in a large magnetic field may yield the spin-degeneracy factor in the thermopower formalism. For this study, quenched  $\text{Fe}_2\text{O}_3$  will be needed. A quenching apparatus has been constructed.

##### Reports:

- No. 1 in previous edition.
2. The Defect Structure of  $\text{VO}_2$ : Comparison Between X-Ray and Electron Diffraction, by M. Morinaga et al, *Acta Cryst A36*, 1070 (1980). AD A095 832
3. X-Ray Diffraction Study of  $\text{Zr}(\text{Ca}, \text{Y})\text{O}_2$ : I. Local Ionic Arrangements, by M. Morinaga et al, *Acta Cryst A36*, 520 (1980). AD A090 769
4. Thermoelectric Study of Cobaltous Oxide Defect Structure, by H. C. Chen et al, *Commun Am Ceramic Soc.*

#### 16822 FORMATION AND PROPERTIES OF POLYMERS USED IN ORGANIC MATRIX COMPOSITES

J. K. Gillham

Princeton University

SL: AMMRC SC: LANGLEY, HDL

The objective of the investigation is to study the relationships among chemical structure, gelation, viscosity, vitrification, and phase separation in determining materials properties of thermosetting systems. The program will include dynamic mechanical analysis, thermal analysis, rheological measurements, scanning and transmission electron analysis, infrared spectroscopy, and stress-strain and impact measurements. An automated torsion pendulum has been installed and chemicals, especially epoxy resins and curing agents have been obtained.

## Reports:

1. TTT State Diagrams of Epoxy-Amine Thermosets, by John B. Enns et al, *Polymer Chem.*

### 16881 NEUTRON DIFFRACTION STUDIES OF SOME RARE EARTH-TRANSITION METAL DEUTERIDES

W. J. James

University of Missouri at Rolla

SL: ETDL SC: MERADCOM

The intermetallic compounds  $\text{LaNi}_5$ ,  $\text{LaNi}_{4.5}\text{Al}_{0.57}$ ,  $\text{Y}_6\text{Mn}_{23}$ ,  $\text{Er}_6\text{Mn}_{23}$ , and  $\text{Ho}_6\text{Fe}_{23}$  have been prepared by the cold boat method using an induction furnace. The samples have been determined to be single phase by the Debye-Scherrer x-ray technique.  $\text{Y}_6(\text{Mn}_{0.6}\text{Al}_{0.4})_{23}$  has also been prepared but evidence of a second phase exists in the x-ray data. A hydrogenation system has been built of stainless steel. It has been tested and used to hydrogenate  $\text{Y}_6\text{Mn}_{23}$  and  $\text{LaNi}_{4.5}\text{Al}_{0.5}$ . Powder neutron diffraction patterns have been obtained and analyzed for  $\text{ErFe}_3$  at 4.2, 77, and 300K, and for  $\text{Er}_6\text{Mn}_{23}$  at 8 and 80K. Powder neutron diffraction patterns have been obtained for  $\text{Y}_6\text{Mn}_{23}\text{D}_{24}$  at 77 and 300K. The nuclear structure at 300K has been determined using the Rietveld profile technique and a X value of 2.0 was obtained. The compound retains the Fm3m symmetry of the non-deuterated  $\text{Y}_6\text{Mn}_{23}$ , with very little change in the positions of the Y and Mn atoms.

## Reports:

1. Neutron Diffraction and Magnetic Studies of  $\text{ErFe}_3$ , by B. Kebe et al, *J Appl Phys.*
2. Magnetic and Structural Properties of  $\text{Y}_6\text{Mn}_{23}\text{D}_{24}$ , by C. Crowder et al, pres 15th Rare Earth Res Conf.
3. Evidence for the Noncollinearity of the Magnetic Structure of  $\text{Er}_6\text{Mn}_{23}$ , by B. Kebe et al, pres 15th Rare Earth Res Conf.
4. Magnetic Properties of  $\text{ErM}_3$ ,  $\text{M} = (\text{Ni}, \text{Fe}, \text{Co})$ , by B. Decrop et al, pres 15th Rare Earth Res Conf.

### 16885 ATOMIC AND ELECTRONIC STRUCTURE OF DEFECTS IN SEMICONDUCTORS

J. Spence

Arizona State University

SC: ETDL, MICOM

Calculations for the electronic band structure of partial dislocations in silicon have been completed.

Dislocations degrade the performance of real devices (particularly light emitting diodes) and their electronic structure provides the key to understanding this degradation. Further calculations for the electronic structure of kinks are in progress. A method of determining the sites of light atom impurities in crystal lattices within nanometer sized regions using energy filtered electron diffraction patterns has been proposed. Since the argument depends only on symmetry, no quantitative data analysis is needed. There are at present no other microanalytical methods for doing this. Near-atomic resolution electron images of interfaces in  $\text{Ga}_x\text{Al}_{1-x}\text{As}$  artificial layer compounds have been recorded and are being analyzed to determine the composition variation across the interface as Al substitutes for Ga. The speed of laser diodes and other devices is reduced by traps at these interfaces, the atomic structure of which are poorly understood. A promising new technique has been devised to allow the chemical environment of selected light atom species to be determined in the lattice from very much smaller areas than is possible using Synchrotron radiation.

## Reports:

1. The Use of Characteristic Loss Energy Selected Electron Diffraction Patterns for Site Symmetry Determination, by J. C. H. Spence, *Optik.*
2. An Exel's Analysis System and Preliminary Orientation Dependence Results, by M. Disko et al.
3. Orientation-Dependent Extended Film Structure in Electron Energy Loss Spectra, by M. M. Disko et al, *Phys Rev Let.*
4. High Resolution Electron Microscopy of Semiconductor Defects, by J. C. H. Spence.
5. The Electronic Structure of the Unreconstructed 30 Degree Partial Dislocation in Silicon, by John E. Northrup et al *Phys Rev.*

### 16911 MECHANISMS OF RADIATION DAMAGE IN FIBER OPTICAL WAVEGUIDES

G. R. Glair

Hughes Research Laboratory

SL: CENCOMS, HDL SC: ETDL

This research program is directed toward a study of the mechanisms generating damage centers leading to decreased signal transmission, when an optical fiber is exposed to ionizing radiation. During the first phase of the program, thermoluminescence (TL) and optical transmission are used as the

diagnostic tools. Doping of the fibers with elements such as Ce, Ga, and Yb is used to examine the feasibility of preventing the formation of color centers which absorb light in the wavelength region of  $0.82\mu$  to  $1.1\mu$ . An aluminum-jacketed, low phosphorus (0.5 mol %)-germania-silica fiber has been examined. The transmission measurements use  $0.82\mu$  light and are made *in situ* during the irradiation. Ten meters of fiber are irradiated in each test. The TL data are obtained at increasing intervals of time between readings, beginning five minutes after irradiation ceases. The TL measurements are made on short lengths (25 cm) of fiber, (rather than powder) using a furnace constructed around a hypodermic needle. The calculation of TL emission kinetics follows the procedure by Von Gorkum. The lathe for preform fabrication will be modified to allow introduction of volatile compounds of Ce, Ga and Yb into the CVD gas stream.

*Reports:*

1. Radiation Damage in Optical Fibers, by G. R. Blair et al.

**17165 STUDIES OF MATERIALS HAVING SIGNIFICANCE FOR HIGH ENERGY PERMANENT MAGNET PRODUCTION AND FOR HYDROGEN STORAGE**

W. E. Wallace  
University of Pittsburgh

SL: ETDL SC: ETDL, MERADCOM

The objectives of the investigation are to study new magnetic materials in hopes of discovering materials of significance for high energy permanent magnets, and to seek better systems for storage of hydrogen. The program will include: *Magnetic Studies:* Synthesis and characterization (magnetic and crystallographic) of  $\text{La}_2\text{Co}_{17}$ ,  $\text{La}_2\text{Fe}_{17}$ ,  $\text{Pr}_2\text{Co}_{17}$ ,  $\text{R}_2\text{Ti}_{12}\text{P}_7$ ,  $\text{R}_6\text{Mn}_{23}\text{-H}$  (where R is a rare earth element and T is a transition element). *Hydrogen Studies:* Measure kinetics of adsorption/desorption and the saturation concentration of hydrogen by  $\text{R}_2\text{Fe}_{17}$ ,  $\text{RAl}_{12-x}\text{Tx}$ ,  $\text{R}_2\text{Ni}$ ,  $\text{CaAl}_2$  and  $\text{TiAl}$ . In studies of  $\text{Er}_{2-x}\text{Pr}_x\text{Co}_{17}$  systems it was found that the anisotropy field (HA) can be incremented from 17 kOe up to 24 kOe by replacing 20% of Er by Pr. Further replacement leads to a decline in HA. The theoretical energy product  $(\text{BH})_{\text{max}} = 34.7 \text{ MGOe}$  for  $\text{Er}_{1.6}\text{Pr}_{0.4}\text{Co}_{17}$ , which is large enough to make

**D. Effects of Structure, Defects, and Composition**

these of interest for permanent magnet fabrication. The enhancement of HA is ascribed to preferential replacement of Er by Pr at the 2d sites. This was established by  $^{166}\text{Er}$  Mössbauer spectroscopy performed on the samples.

**17215 THE CEMENT-AGGREGATE AND ITS INFLUENCE ON CONCRETE DURABILITY**

Jan P. Skalny  
Martin Marietta Laboratories  
SL: MICOM, WES SC: MERADCOM, CRREL

The objective of this investigation is to study the chemical morphological and mechanical nature of the cement aggregate bond and how it is affected by aggressive solutions with the aim of improving the resistance to degradation. The program will include the following tasks: (1) Characterize the morphology, chemistry and mechanical properties of the cement aggregate bond using electron microscopy, spectroscopic techniques, permeability, strength and hardness measurements. (2) Determine the role of the cement aggregate bond on concrete durability by evaluating the effect (using above techniques) of various aggressive solutions on selected, well-characterized concrete samples. (3) Use information from first two tasks to modify the chemical nature of the bond to improve bond strength and durability.

**17298 BARRIERS TO THE MIGRATION OF INTERPHASE BOUNDARIES**

Hubert I. Aaronson  
Carnegie Mellon University  
SL: AMMRC, BWL SC: MERADCOM, CERL, TARADCOM

Research will be conducted on boundaries to the migration of interphase boundaries, especially in steels. The work will include: (1) Investigation of incomplete transformation in high purity Fe-C-X alloys ( $X = \text{Si, Ni, Co, Mo, Cr, V, and Ti}$ ) with emphasis on acquiring a theoretical understanding of these phenomena. (2) Investigation of interfacial structure to ascertain whether martensitic plates thicken by shear or by diffusional growth via a ledge mechanism.



**17546 ELECTRICAL, CHEMICAL, AND MICROSTRUCTURAL CHARACTERIZATION OF GETTERING MECHANISMS FOR VLSI**

J. Stach  
J. R. Monkowski  
I. S. T. Tsong  
Pennsylvania State University

The objective of the investigation is to study the basic mechanisms whereby several gettering techniques interact with defects in silicon to produce "defect-free" silicon. The program will include: (1) Characterization of the defects (vacancies and interstitials, dislocations, stacking faults) in several starting single crystals, of silicon using transmission electron microscopy, sputter-induced photon spectroscopy, secondary ion mass spectroscopy, and thermally stimulated current. (2) Application of gettering techniques, e. g., backsurface damage, intrinsic gettering, chlorine oxidation, diffusion, annealing. (3) Characterization of the "gettered" material and measurement of carrier lifetimes.

**17716 CONTROL OF ELECTRONIC, MAGNETIC AND OPTICAL PROPERTIES OF DOPED POLYACETYLENE MATERIALS THROUGH INDUCED DEFECTS**

Alan J. Heeger  
Shahab Etemad  
University of Pennsylvania  
SC: NVEOL, MERADCOM, FCSCWSL

The objective of this research is to study the effects of radiation induced defects on the optical, magnetic and electronic properties of doped polyacetylene conductors. The approach will be to characterize defects in polyacetylene,  $(CH)_x$ , systems doped with donors or acceptors in the semiconducting or metallic regimes. The research will include electron spin resonance and magnetic susceptibility measurements, DC electrical conductivity and thermopower measurements microwave conductivity and dielectric constant measurements, optical studies including reflection, transmission and Raman spectroscopy, and ion implantation doping studies.

**Reports:**

1. Photoexcitations in Polyacetylene, by L. Lauchlan et al, *Phys Rev.*

**17790 FERROMAGNETIC FLUIDS**

W. Funderburk  
Barber-Scotia College  
SC: ETDL

The objective of the investigation is to study the particle size, particle coagulation, fluid stability, and magnetic anisotropy of both electrically conducting and non-conducting ferromagnetic fluids. The program will include: (1) Determination of the effects of ultrasound in the frequency range 1-30 MHz at room temperature in the presence of external magnetic fields upon both conducting and non-conducting ferrofluids. (2) Theoretical consideration of magnetic anisotropy and particle size and state of agglomeration in such ferrofluids.

**17942 THE RELATIONSHIP BETWEEN RADIATION SENSITIVITY AND REDOX EQUILIBRIA**

D. L. Kinser  
R. A. Weeks  
Vanderbilt University  
SL: CENCOMS SC: HDL

The objective of the investigation is to determine the relationship between oxidation/reduction equilibria in glasses and the performance of the glass as an optical waveguide in radiation environments. The program will include: (1) Measurement of the optical loss in glasses as a function of thermal history, state of oxidation, and energetic phonon dose. (2) Characterization of the defects in the glasses using optical absorption currents. (3) Formulation of theoretical models to relate the effects.

**17973 STUDY OF THE MAGNETIC AND METALLURGICAL PROPERTIES OF  $Sm(Co,Fe,Cu,Zr)_x$  ALLOYS**

K. J. Strnat  
A. E. Ray  
University of Dayton  
SL: ETDL SC: MERADCOM

The objective of the investigation is to study the magnetic and metallurgical properties of  $Sm(Co,Fe,Cu,Zr)_x$  alloys. The program will include: (1) Preparation of the alloys, to include

induction melting, casting, solution and aging heat treatments. (2) Chemical analysis of the alloys. (3) Metallurgical characterization of the alloys, including electron microscopy, optical microscopy, x-ray diffraction. (4) Magnetic characterization of the alloys, including magnetization vs. applied field,  $4\pi M_s$ , anisotropy field, intrinsic coercivity.

#### E. NEW CONCEPTS IN TESTING, ANALYSIS, AND SIMULATION

##### 15746 LASER PHOTOACOUSTIC TECHNIQUE FOR NDE

R. L. Thomas

Wayne State University

SL: BRL SC: MERADCOM, EUSTIS, TARADCOM

The SPAM technique has been extended from ceramic materials to metals, and the theory of subsurface defect detection in a well-characterized Al sample has been carried out, with detailed agreement with experiment. SPAM techniques have been developed for detecting surface and subsurface flaws in ceramic turbine engine parts of complex geometry, and a variety of such flaws have been successfully detected in  $\text{Si}_3\text{N}_4$  ceramic stator vanes. This work is currently being extended to SiC samples. The SPAM technique has also been microprocessor-controlled, with digital data acquisition. This has permitted the first true photoacoustic microscope pictures to be taken of an integrated chip, with  $6\mu\text{m}$  resolution. Studies have also been initiated of subsurface damage associated with ion-implantation of boron in silicon wafers.

#### Reports:

1. Scanning Photoacoustic Microscopy - NDE, by J. J. Pouch et al.
2. Subsurface Structures of Solids by Scanning Photoacoustic Microscopy, by Y. H. Wong et al. *Appl Phys Lett* 35, 368 (1979).
3. Subsurface Flaw Detection in Metals by Photoacoustic Microscopy, by R. L. Thomas et al. *J Appl Phys* 51, 1152 (1980). AD A085 374
4. Scanning Photoacoustic Microscopy for Nondestructive Evaluation, by J. J. Pouch et al. *J Opt Soc Am* 70, 562 (1980). AD A092 490
5. Photoacoustic Microscopy of an Integrated Circuit, by L. D. Favro et al. *Appl Phys Lett* 36, 953 (1980).
6. Photoacoustic Microscopy of Ceramic Engine Hardware, by L. I. Inglehart et al.
7. Photoacoustic Microscopy, by L. D. Favro et al. *Proc of DARPA/AF Rev of Progress in Quantitative NDE*.

##### 16964 CLUSTER VARIATION STUDY OF COHERENT ORDERING IN FCC AND BCC SOLID SOLUTIONS

D. de Fontaine

University of California at Berkeley

SC: BWL

A study is being made of the effect on the phase diagram of the ratio of first to second neighbor pair interaction energies for a prototype Ni-V binary system. The primary objective is to determine the smallest set of parameters which are essential to determine the types of alloy phase diagrams experimentally observed, and how small changes in the values of these parameters alter the shapes of phase regions. Interesting applications of these calculations to the problem of phase relations in Ni-based superalloys is foreseeable. A fluctuation theory has been developed which is closely related to the Cluster Variation Method used for the phase diagram calculation. Among the most interesting aspects of the theory is that one can compare measured experimental short-range order x-ray diffuse intensity with results obtained from the Cluster Variation Method. Thus, for the first time, the theoretical tools are available for a complete and consistent study of binary solid solutions in a very reliable approximation.

#### Reports:

1. Special Point Ordering in General Crystal Structures, by J. M. Sanchez et al. *Acta Cryst A*.
2. Compositional Instabilities, by D. de Fontaine.

##### 17923 MAGNETIC FIELD INTERACTIONS WITH MATERIAL DISCONTINUITIES

W. Lord

Colorado State University

SL: AMMRC, MICOM, ETDI

The objective of the research is to develop finite element analysis techniques which can be applied to the problem of modeling electromagnetic nondestructive testing phenomena. The approach is to develop finite element analysis techniques to overcome the awkward defect boundary shapes and nonlinear material characteristics encountered in electromagnetic nondestructive testing. In-depth studies have been made of active and eddy current excitation modes. This proposal seeks to extend the technique to residual leakage fields and pulsed eddy current phenomena.

**18220 HIGH FREQUENCY QUANTITATIVE  
ANALYSIS OF  
MAGNETOMECHANICAL ACOUSTIC  
EMISSION**

Kanji Ono

University of California at Los Angeles

SC: MICOM, TACOM, AMMRC

The objective of the research is to investigate the use of magnetomechanical acoustic emission as a

means of nondestructively measuring residual stresses. The magnetomechanical acoustic emission (MAE) is a complex function of chemical composition, microstructure, prior cold work, stress state and external magnetic excitation. The proposed research hopes to extract more definitive information as to how MAE relates to internal magnetic structures using quantitative signal analysis methods and by extending the frequency range to 50 to 100 MHz.

## V EUROPEAN RESEARCH PROGRAM

### A&B. CHEMISTRY AND BIOLOGICAL SCIENCES

#### 2376 THE INFLUENCE OF LATTICE IMPERFECTIONS ON THE CHEMICAL REACTIVITY OF SOLIDS

J. N. Sherwood  
University of Strathclyde

SL: ARRADCOM SC: AMMRC, BRL

The basic dislocation analysis in RDX has been completed and effect of temperature during crystal growth on plastic deformation assessed. The work has been extended to direct deformation studies via microhardness measurements. Satisfactory crystals of PETN,  $\beta$ -HMX, TNT, DATB and nitroguanidine have been grown and will be studied by X-ray topography.

#### 2522 ANALYSIS OF STEROIDS BY OPEN-TUBULAR GC-MS

C. J. W. Brooks  
University of Glasgow  
SL: NLABS, LAIR SC: LAIR

The research has been divided between preparative steroid chemistry and gas chromatography. The sterol (25R) -26-hydroxycholesterol was prepared by partial synthesis from the steroid sapogenin, diosgenin. Samples of  $7\alpha$  - and  $7\beta$  hydroxycholesterol were also prepared. Gas chromatography based on packed columns have been used in conjunction with selective enzymic oxidations in the separation and characterization of sterols. The use of open tubular pyrex columns is being studied for the efficient separations of sterols (as their trimethylsilyl derivatives).

#### 2687 HAPTEN IMMUNOLOGICAL STUDIES OF TOXIC ORGANOPHOSPHATES

A. Kalir  
S. Ben Effraim  
Sackler School of Medicine

The specific aim of this research was to devise conditions for preparation of antibodies against several congeners of pinacolyl phosphonic esters and of pinacolyl alcohol, on the assumption that such antibodies will react with the chemically related toxic organic phosphonofluoridate soman.

The following compounds were synthesized and characterized: pinacolyl oxypropionic acid ( $C_1$ ); pinacolyl (1,1,2-trimethoxypropyl) 4-amino-benzoate ( $C_2$ ); methylphosphonyl dichloride, pinacolyl methylphosphonochloridate and the dicyclohexylamine salt of O-pinacolyl methylphosphonic acid. Conjugates of  $C_1$  or  $C_2$  with rabbit serum albumin or human serum albumin were prepared and used for immunization of rabbits. Conjugates of  $C_1$  or  $C_2$  with lysozyme were used in enzyme immunoassays.

Antibodies against  $C_1$  and  $C_2$  compounds were produced in rabbits as shown by hemagglutination, complement fixation and enzyme immunoassays for  $C_1$  compound and by enzyme immunoassays for  $C_2$  compound.

#### 2693 ANODIC PROCESSES IN THE LITHIUM/SULFURYL CHLORIDE BATTERY

B. Scrosati  
University of Rome  
SL: ETDL SC: HDL

Preliminary results indicate that the lithium passivating film in sulfuryl chloride batteries consists of two layers. The inner layer, formed immediately upon contact with the electrolyte, is



probably very thin and compact. The outer layer, which keeps growing on top of the first, is probably thick and very defective. The activation of the electrode upon discharge occurs through the cracks and channels in this layer. Current results on the polarization of the lithium electrode in such systems under a variety of conditions are consistent with this model.

#### 2696 ALKANE-SOLUBLE TRANSITION METAL COMPLEXES

F. R. Hartley  
Royal Military College of Science  
Shrivenham  
SL: CSL SC: NLABS

Two series of tertiary phosphine (a trialkyl- and a triaryl-) ligands that confer unusual solubility properties on their metal complexes have been prepared. Complexation with Pt, Pd and Rh salts resulted in five series of complexes, all of which were found to be exceedingly soluble in saturated hydrocarbons and in chlorinated solvents. They were moderately soluble in aromatics, alcohols, acetone and in other ethers, thus exhibiting the same solubility characteristics as the parent phosphines.

#### 2741 REACTIONS OF METASTABLE RARE EARTH GASES WITH COPPER ATOMS

S. Rosenwaks  
Ben Gurion University  
Beer Sheva  
SC: MICOM

The interaction of metastable rare gases with copper atoms has been studied by emission and absorption spectroscopic techniques. Emission was observed from neutral Cu atoms in a variety of electronic states. In the reaction with excited He, emission from Cu<sup>+</sup> states lying up to 13.7 eV above ground state Cu<sup>+</sup> was observed. It was found that Ne efficiently quenched the lower lying Cu<sup>+</sup> states. The concentration of Cu atoms in the ground state was measured by atomic absorption, using a Cu hollow cathode lamp.

#### 2792 RARE EARTH GLASSES AS LIGHT SOURCES

R. Reisfeld  
Hebrew University  
Jerusalem  
SL: CECOM SC: ETDL, NVEOL

Spectroscopic studies were made on a neodymium-doped gallium lanthanum sulfide glass. It was found that a sample containing 0.5 weight percent Nd<sub>2</sub>S<sub>3</sub> had a fluorescence decay time of ca. 70 μsec. A photoconducting PbS crystal infrared detector has been purchased and installed in the spectrometer that will extend the spectral sensitivity of the instrument out to 2 microns. This permitted the observation of a fluorescent emission of Nd<sup>3+</sup> at 1.35 μm for the first time.

#### 2794 BIMANE DERIVATIVES AS FLUORESCENT PROBES FOR BIOLOGICAL MACROMOLECULES

E. N. Kosower  
Tel Aviv University  
SC: NLABS, CSL

When a promising "one pot" procedure for the synthesis of vinylbimanes was found to give very poor yields, it was decided to prepare these compounds by an alternate route, involving bromination followed by the elimination of triethylamine. These compounds are bright yellow, presumably due to intermolecular interactions in the solid state. The vinyl group appears to be strongly conjugated with the chromophore in the SI state (but not in the ground state), resulting in a red shift of their fluorescence. Two vinylbimanes were found to react with red blood cells, attesting to their potential as fluorescent probes.

#### 2798 SOLUBILIZATION AND REACTIONS IN COLLOIDAL AGGREGATES

M. Graetzel  
Ecole Polytechnique Federale de Lausanne  
SL: CSL SC: NLABS

Several new cationic microemulsions have been developed and characterized by their phase diagrams. Their solubilizing capacities with respect

to tetrathiofulvalene have been measured. In addition, a number of their physical parameters (viscosity, pH, density, refractive index and hydrodynamic radius) have been measured. In order to obtain information about the dynamics and micro-viscosity of the interior and the local polarity of the aggregates, interaction of these emulsions with a variety of substrates have been investigated by means of fluorescence, laser-flash spectroscopic, light scattering and NMR techniques.

### 2813 DEFORMATION, FRACTURE AND EXPLOSIVE PROPERTIES OF REACTIVE MATERIALS

J. E. Field  
M. M. Chaudri  
University of Cambridge  
SL: LCWSL

The proposed research involves studies on the mechanical, the thermal and the explosive properties of reactive materials. The techniques developed in earlier work for taking high-speed photographs and pressure/time records during drop-weight impact will be used for experiments on RDX, TNT, HMX, HNS, TATB, HQ and binders. The effect of additives on drop-weight sensitiveness will be studied. A variety of techniques, already under development, will be used to investigate the fracture, flow and strength properties of both single crystals of explosive and explosive/binder systems. Finally, there will be research on the acquisition and analysis of decomposition data, with the objectives of understanding the mechanisms of decomposition and making the data recording and processing as automatic as possible.

### 2817 MASS SPECTROMETRY OF EXPLOSIVES MIXTURES IN WATER

J. Yinon  
Weizmann Institute  
Rehovot  
SL: LCWSL, USATHAMA SC: NLABS, MERADCOM

Explosives traces in water were analyzed by direct injection chemical ionization (CI) mass spectrometry, using the endogenous water as CI reagent. Investigated explosives for trace analysis

were p-nitrotoluene, 2,4-DNT, TNT, m-DNB, a mixture of TNT and TNB, tetryl, RDX, HMX and PETN. Using partial scanning and a strip chart recorder, minimum amounts of explosives detected were in the range of 2-40 ng. A reduction process was found to occur in the ion source when observing the CI mass spectra of a series of trinitroaromatic compounds, using water as reagent. The  $(MH - 30)^+$  ions in the CI mass spectra were mainly due to the reduction of the investigated trinitroaromatic compounds to their corresponding amines. This was proved by using  $D_2O$  as reagent: the  $(MH-30)^+$  ions were shifted to  $(MD - 28)^+$  ions. The investigated trinitroaromatic compounds included TNB, TNT, TNC, picramide and picric acid.

### 2845 CONFORMATIONAL FLUCTUATIONS IN PROTEINS BY USE OF OPTICAL PROBES

J. Feitelson  
Hebrew University  
Jerusalem  
SL: AMRDC SC: NLABS

A temperature-dependent spectroscopic study of the excited state interaction of 3-methylindole with ethyl acetate indicates exciplex formation over a temperature range down to  $-50^\circ C$  and groundstate complex formation at lower temperatures. Work is in progress on the quenching of the phosphorescence of alcohol dehydrogenase (from horse liver) by oxygen, as a function of temperature.

### 2865 THERMOLYTIC INTERCONVERSION OF BORON HYDRIDES

N. N. Greenwood  
University of Leeds  
SL: MICOM

The inlet system to the reaction chamber was upgraded to permit molecular flow in the capillaries and a Kratos DS50CO data system coupled to the MS30 mass spectrometer to improve the processing of the results. In addition, an MS10 instrument was coupled to the MS30 to allow for the continuous monitoring of  $H_2$ , HD and  $D_2$ . Preliminary

evidence indicates that at temperatures of 50-75°C  $B_6H_{10}$  is in equilibrium with  $B_4H_8$  and  $H_2$ . They also found that  $B_6H_{10}$  was stable at 75°C, but loses  $H_2$  at 120°C. At 75°C  $B_6H_{10}$  will interact with other boranes by an as yet unknown mechanism.

#### 2874 NS TIME-RESOLVED FLUORESCENCE STUDIED IN ENERGY MIGRATION IN SYNTHETIC POLYMERS

D. Phillips  
The Royal Institution  
London  
SC: AMMRC, NLABS

Considerable progress has been made in the understanding of excimer formation and decay in vinyl aromatic polymers and in the photopolymerizable polyesters of p-phenylene diacrylic acids. For copolymers of l-vinyl naphthalene with methyl methacrylate the rate constants for the polymer photophysical processes were determined at different temperatures. Quenching studies gave evidence for a long-range energy transfer process. Poly (N-vinyl carbazole) exhibits no monomer fluorescence, but emissions due to the formation of three excimer species. The polyesters of p-phenylene diacrylic acid show both monomer and excimer emissions, the latter appear to be pre-formed in the ground state.

#### 2876 SUGAR OXIMES: POTENTIAL ANTIDOTES AGAINST ORGANOPHOSPHORUS POISONING

E. S. Rachaman  
Israel Institute for Biological Research

This research will concern the bio-availability of sugar oximes in the body and on their capability to penetrate the blood-brain barrier.

##### Progress:

Preliminary pharmacological studies have indicated that some of the sugar oximes are more potent than non-sugar analogs against organophosphorus (OP) poisoning. In parallel to

this research direction, we shall attempt to improve our understanding of the structure activity relationship in this series of compounds.

Seven new sugar oximes derived from 3- or 4-pyridinealoximes by quaternary attachment to the ring N were synthesized and tested as potential antidotes for organophosphorus inhibited AChE. Several showed positive antidotal properties, two were particularly promising with less apparent side effects than the parent methiodides. Test anticholinesterases were paraoxon and VX. Future work will include derivatives of 2-PAM and TMB-4. Studies of efficacy as a prophylactic drug alone and in mixtures with other drugs is in process.

To date, studies indicate that the sugar moiety does not change significantly the distribution coefficient between octanol and aqueous buffer. The correlation between *in vitro* enzyme reactivation and antidotal efficacy is poor. Thus, the mechanism for observed protection continues obscure.

#### 2890 CELLULAR MECHANISMS OF CENTRAL NERVOUS MODULATION

John E. Treherne  
Cambridge University

A new project to investigate the cellular mechanisms involved in central nervous modulation by biogenic amines. The observed effects of octopamine on the insect perineurium provides a novel opportunity to study aminergic modulation of glial function using sucrose-gap recording, conventional and ion-selective microelectrodes, radioenzymatic assay and ultrastructural techniques. It is also proposed to test the effects of a range of pharmacological compounds on the superficial neuroglia, to investigate their effects on cellular permeability and the properties of the blood-brain barrier. The modulation of central nervous synaptic transmission by octopamine will also be studied by exploiting the advantages provided by the sabellid giant axon preparation. This will involve measurement of the effects of aminergic drugs on miniature end plate potentials, identification of the synaptic pathways and neurochemical investigations.

**3047 REACTIVE INTERMEDIATES IN  
ENERGETIC SYSTEMS**

B. A. Thrush  
University of Cambridge  
SL: LCWSL SC: BRL

The primary aim of this research is the direct measurement of the rates of reaction of HNO with H, OH, HNO and NO. These reaction rate coefficients would be measured initially over the temperature range 260-360 K. On the basis of these studies, attempts would be made to detect HNO in the pyrolyses of alkyl nitrites, nitrates and nitrocompounds and study its reactions in such systems. These investigations should indicate which other reactions of HNO are important in the decomposition of energetic materials.

**C. MECHANICS AND AERONAUTICS****2526 ON THE MANIPULATION OF THE  
MIXING PROCESS BETWEEN TWO  
STREAMS OF DIFFERENT VELOCITY  
AND TEMPERATURE IN THE  
PRESENCE OF A PRESSURE  
GRADIENT**

S. Einav  
Tel-Aviv University  
SL: MICOM SC: MICOM

The mechanism of growth of the two dimensional turbulent mixing region is not completely understood in spite of the fact that the flow has been continuously investigated during the last decade. A detailed discussion of the views held by many researchers on turbulent mixing is presented in a volume edited by Murthy (1975) to which the reader may refer. It is becoming increasingly evident, however, that the flow in the turbulent mixing layer is dominated by a row of large vortices which retain their basic structure over long distances. The individual vortices in the row grow as they proceed downstream, but the overall spread of the mixing layer is attributed largely to the interaction between neighboring vortices which roll around each other to form larger coherent structures. The study will focus on systematically examining the effects of initial flow conditions, pressure gradient, and mechanical disturbances on the spreading of the mixing layer and turbulence

intensity. The experiments will be conducted in a two stream wind tunnel, utilizing a range of Reynold's numbers. The study will then be repeated in a water tunnel. Flow visualization technique will be used to determine the mechanism(s) which can be used to control amalgamation of large eddy structures in the mixing layer and hence the mixing process.

**2600 THE FATIGUE CRACK GROWTH  
UNDER VARIABLE AMPLITUDE  
LOADING IN BUILT-UP STRUCTURES**

A. Salvetti  
Universita degli Studi di Pisa  
SL: AMMRC SC: ARRADCOM, EOARD

Research aimed at developing reliable methods for predicting the growth of a crack in built-up aircraft structures under realistic load conditions is currently being carried out at the Institute of Aeronautics of the University of Pisa under a three year research contract which is jointly sponsored by the European Research Office and the European Office of Aerospace Research and Development. In the course of the second year, 33 specimens were tested both at constant and variable amplitude loading. The specimens, cut from sheets of 7075-T6 aluminum alloy, coming from the same batch, were both simple sheets and riveted, stiffened panels. The constant amplitude tests on simple sheet specimens were carried out in order to obtain the average K-rate relationship and the relevant scatter of the batch of sheets. The constant amplitude tests on stiffened panels were aimed at obtaining information on rivet flexibility and friction forces. Variable amplitude tests were performed utilizing the FALSTAFF spectrum. The data on sheet specimens was used to assess the reliability of prediction methods such as those devised by Wheeler and Willenborg, and the stiffened panels test data to establish how these methods work in the case of built-up structures. The results presented so far, especially those concerning the spectrum tests on stiffened panels, are still preliminary, but significant in as far as they allow us to assess the reliability of crack growth prediction methods and to obtain a qualitative but deep insight into the phenomenon of crack growth in built-up structures. The program of further tests, planned for the third year, has been drawn up with a



view to the quantitative assessment of the complex phenomenon described above.

### 2639 BREAK-UP OF VORTEX RINGS IN IMPINGING TURBULENT JET FLAMES

N. Chigier  
University of Sheffield  
SL: LEWIS SC: TACOM, AFLRL

The objective of this experimental investigation is to examine the structure of turbulent flow in jet flames impinging on flat walls with particular emphasis on the formation and breakup of vortex ring-like eddies. Experiments include the study of heat transfer to the wall, and the influence of eddies on the mechanism of heat transfer, with variation in flow geometry and burner initial conditions. During the first year of research a new flame-impingement rig has been commissioned, preliminary flow visualisations have been made showing flame structure over a wide range of conditions. Considerable data have been obtained in non-impinging flames, which will later be compared with impinging flame data. As a result of this preliminary work it is found that ring vortex eddies are identified in impinging flames for a wide range of flow geometries, equivalence ratios and types of gaseous fuel. These eddies break up into 'flamelets' which remain coherent for long distances. These coherent structures require further investigation because they must strongly influence heat transfer to the wall and fluctuations in gas temperature near the wall. Similar structures have been identified and measured in non-impinging flames. Following the completion of the measurements with ionization probes, mean concentration of stable species was measured across the flames using sampling probes and on-line analysis for CO, CO<sub>2</sub>, HC, and O<sub>2</sub>. These mean concentration measurements provide the overall characteristics of changes in composition of major species. They provide information on the rate of consumption of fuel and on the rate of entrainment of air. Thermocouple measurements have been completed yielding mean, rms, pdf, spectra, and correlations. Joint measurements of velocity and temperature have been performed, enabling the calculation of the important correlation,  $u'p'$ . In addition, a rake of eight thermocouples has been constructed and used, enabling average temperature profiles in eddies to be measured.

Sections of the eddies where there are flame fronts are detected on each probe as peaks, which can then be correlated to provide information on eddy structure.

### 2640 BREAK-UP OF VORTEX RINGS IN IMPINGING QUASI-ISOTHERMAL JETS

J. Mathieu  
Ecole Centrale de Lyon  
SL: LEWIS SC: TACOM

The proposed experimental study is concerned with the coherent structures of a round impinging jet. This flow is quasi-isothermal. Slight heating is used for tagging the structures. Attention will be focused on the formation and breakup of vortex rings. By means of conditional sampling (zone or point averages) the fluid properties on either side of free interfaces will be determined. By injection of seeded particles of heat pulses the motions of an isolated structure will be examined. The detection probes will be hot wires and a laser doppler anemometer.

### 2689 THE CURRENT STATUS OF BLAST WAVE THEORY AND COMPUTATION TECHNIQUES

M. M. Kamel  
University of Cairo  
SL: ARRADCOM SC: BRL

During three and half decades of blast wave theory research, the number of published articles, reports, review papers and books has mushroomed tremendously. As the volume and sophistication of the published work increases, it becomes harder for newcomers to the field, even for old hands, to sort out the different methods, nomenclatures and conclusions of different authors. This proposed program of research, then, intends to critically review blast wave theory research from the early forties to the present, with emphasis on recent developments. For the sake of continuity and clarity, this exposition will endeavor to present a unified formulation of the subject, with solutions to specific problems by various authors discussed and evaluated. In discussing the methods of solution, emphasis will be given to both numerical and

analytical solutions of particular problems. The range of validity of each method of solution and the feasibility of its utilization to solve other problems will also be evaluated.

## 2690 SPRAY MODELLING FOR MULTIFUEL ENGINES

M. M. Elkoth  
University of Cairo  
SC: AFLRL, TACOM

The present project deals with fuel spray modelling in multifuel engines using the droplet behavior approach. The multifuel engine is able to burn different types of fuel having various physical and chemical properties, either clear or mixture with arbitrary mixing ratio. Analysis of the physical and chemical properties affecting mixture creation and combustion have been carried out to define the parameters affecting the accuracy of spray modeling. It is found that the spray is effected by air flow field, heat transfer, fuel atomization, droplet behavior, fuel evaporation and chemical kinetics. Solution of the flow field and experimental results of heat transfer have been obtained. Experiments were performed in a setup that allowed variation of the swirl chamber geometry and operating conditions to evaluate the instantaneous surface heat transfer under firing conditions. The instantaneous heat flux was determined taking into consideration the instantaneous composition of products and variation of specific heats. A generalized expression has been established for the calculation of heat transfer to the chamber wall under variable design and operating conditions. Prediction of the air flow field is based on the solution of the finite difference analogue of the governing differential equations for the transport of mass, momentum and energy. The  $K-\epsilon$  model of turbulence has been used. A special technique has been established to satisfy compressibility, local continuity and reduce the mass source at each grid node to zero. Measurements of air flow field are carried using a hot wire anemometer. Agreement between predictions and measurements is fairly good, which confirms the accuracy of the prediction procedure.

## 2736 ANALYSIS OF PARAMETRICALLY EXCITED LARGE VIBRATION SYSTEMS

G. Schweitzer  
Eidenossische Technische Hochschule  
Zurich  
SL: LANGLEY

In many technical systems, the parametrical excitation can be considered as a small periodic perturbation of a system with constant coefficients. Denote  $f$  the number of degrees of freedom of the system,  $\epsilon$  the amplitude, and  $\Omega$  the fundamental frequency of the parametric excitation. The objective of the investigation presented here is the calculation and plot of the stability chart in an effective way. First of all the stability of the constant system ( $\epsilon = 0$ ) is analyzed. By the necessary conditions of parametric resonance of order  $n$ ,  $f^2$  possible critical frequencies are obtained. A frequency is actually critical only when a wide instability region emerges for increasing  $\epsilon$ . Therefore the stability is investigated in the neighborhood of the possible critical frequencies by numerical analysis. If unstable points are detected, the investigated frequency is actually critical. The direction of the stability boundary starting from the critical frequency is determined by a simple method. This information is used to start a boundary tracing algorithm. Therefore, two tracing strategies were developed, namely predictor-corrector method with linear or circle extrapolation and triangulation method. The procedures are applied on a simplified model of the two-bladed wind turbine with three degrees of freedom. It results that among nine possible critical frequencies only two are indeed critical: a principal resonance and a sum type combination resonance occur. The method derived is sufficiently effective to calculate systems of higher order. It will be used as a reference method for further research plans.

## 2835 TURBULENCE STRUCTURE IN UNSTEADY BOUNDARY LAYERS AND WAKES IN OSCILLATING AIRFOIL

C. Hirsch  
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SL: LANGLEY, AMES

The main objective of the present study is to provide experimental information about

turbulence structures in unsteady flow fields. Helicopter blade behavior, turbomachine blade row interactions and marine propeller flows are typical application fields of unsteady phenomena, where a correct understanding of the detailed unsteady flow and turbulence structure is important for predicting overall flow properties. Detailed measurements are hence to be made in time as well as in space in the complete flow field around the considered oscillating blade. In the present study, the blade is a NACA 0012 airfoil which oscillates around an axis at 25% chord from the leading edge, with a sinusoidal motion. A slanted hot wire anemometer technique combined with a periodic sampling system is used for the turbulence measurements in the time domain. Measurements were first performed in the steady state wake of the airfoil at  $0^\circ$ ,  $5^\circ$  and  $9^\circ$  incidence. In the second phase, measurements in the wake of the airfoil were performed around the same mean incidences but with an oscillation amplitude of  $\pm 5^\circ$  and at two frequencies (a low and a high value). These measurements include the mean flow and Reynolds stresses variables. All measurements were done at 5% chord distance from the trailing edge.

#### 2872 INELASTIC DEFORMATION OF METALS AND STRUCTURES UNDER DYNAMIC AND QUASI-STATIC CYCLIC LOADING

A. M. Eleiche  
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SL: ARRADCOM

Research is proposed on the inelastic deformation of metals and structures under both dynamic and quasi-static cyclic loading conditions. The proposed strategy of research is: a. Carrying out critical experimental material tests in order to understand the basic material behavior. b. Using this basic material data, more faithful constitutive relations than those of classical plasticity theories will be developed. c. Using these constitutive relations, analyses will be performed to obtain more acceptable predictions of the response of simple structural components under given loading conditions. Interaction between experimentation, representation and computation will be kept in mind at all stages of the work.

#### 2885 ENGINEERING MODELS OF BALLISTIC PERFORATION AT HIGH VELOCITIES

S. Bodner  
Technion - Israel Institute of Technology  
SL: BRL SC: AMMRC

The actual physical process of perforation is extremely complex and the analysis attempts only to identify the main features of the process and thereby to formulate the governing equations of motion. In the analysis, the perforation process is considered to consist of three interconnected stages, namely, an initial compression followed by combined compression and formation of a plug and finally the shearing of a plug until the target material fails and the plug is ejected. Among the factors neglected in the analysis are elastic and plastic wave effects and compressibility of the target material. For these tests, the projectiles were cylindrical rods of 4340 steel which impacted the target plates perpendicularly at velocities in the range of 1350 to 1550 m/s. Most of the target plates were of rolled homogeneous armor steel and a few were of 5083-H 115 aluminum alloy. Of the total of 10 tests which were performed, 6 on the RHA and 2 on the aluminum targets were reasonably successful. Flash x-ray photographs taken during the successful runs provide information on the initial and residual velocities and on the post perforation geometries of the projectiles. In a second series of testing tungsten projectiles will be applied to thicker targets. On the analytical side, a theoretical model for ballistic perforation was formulated that accounts for the effects of erosion of the projectile and of the target, and also for plastic deformation of the target. The equations for the analytical model are now in a form suitable for computer programming which will be carried out during the next phase of the program.

#### 2887 VELOCITY CHARACTERISTICS OF THE FLOW IN MOTORED ENGINES

J. Whitelaw  
Imperial College of Science and Technology  
SC: TACOM

Measurements of mean and rms values of axial velocity, obtained with a laser anemometer, are

reported in a piston cylinder assembly motored at 200rpm, in the absence of compression. The inlet arrangement consisted of a simulated open valve inclined at  $60^\circ$  to the cylinder head and incorporated  $30^\circ$  swirl vanes which provided a swirl number of about 1.2. Results, obtained with a flat piston and a 90mm stroke, are compared to those with a 60mm stroke reported elsewhere. In addition to the bore to stroke ratio influence, the influence of clearance volume is investigated by comparing results for two different clearance distances of 30mm and 10mm. The results show that longer stroke causes the formation of stronger vortices while smaller clearance generates higher turbulence levels at the early part of the intake stroke; in both cases vortices decay as the piston approaches BDC. Measurements of swirl velocity are also reported in a single-cylinder Diesel engine motored at 675 rpm to quantify the influence of compression. The results show close correspondence with the noncompressing results at 500 rpm in the intake stroke and the beginning of the compression stroke. At higher crank angles the flow tends towards solid body rotation. The experimental results in the noncompressing engine are compared with calculations obtained by numerical solution of the conservation equations and show that the major flow features are correctly predicted.

### 3012 FRACTURE BEHAVIOR UNDER IMPACT

G. Kalthoff  
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SL: BRL SC: ARRADCOM

An experimental investigation is proposed to determine the dynamic stress intensification at the tip of a crack in specimens which are loaded dynamically by impinging projectiles. The shadow optical method of caustics will be utilized in combination with high speed photography for measuring the dynamic stress intensity factors and the passage of the stress pulse through the specimen. These data will be supplemented by strain gage information. In particular, the fracture toughness value corresponding to the onset of crack propagation and the fracture response time will be determined. Initial crack lengths and striking velocities will be varied throughout the experiments. The results of these tests will be applicable for the prevention of failure in the design

of commercial and military structures and devices subjected to transient loading. Utilizing the shadow optical method of caustics in combination with high speed photography the dynamic stress intensity factors,  $K_{I\text{ dyn}}$ , at the tip of the crack will be measured as a function of time during the impact event. The passage of the stress pulse through the specimen will be monitored by strain gages. In addition the stress pulse will be qualitatively made visible by the shadow optical measuring technique. The dynamic stress intensity factor results,  $K_{I\text{ dyn}}(t)$ , will be discussed with respect to the applied stress pulse,  $p(t)$ . In particular, the critical value of the dynamic stress intensity factor at the onset of rapid crack propagation, i. e. the dynamic fracture toughness  $K_{I\text{ d}}$ , will be determined and compared to the corresponding static fracture toughness  $K_{I\text{ c}}$ . Fracture response times will be deduced from the measured data.

### 3017 BURSTS AND PRESSURE FLUCTUATIONS IN TURBULENT BOUNDARY LAYERS

P. Bradshaw  
Imperial College of Science and Technology  
SL: MICOM SC: AMES

Basic work in low-speed wind tunnels is proposed, to investigate the behavior of "bursts" (regions of intense turbulent activity) in the inner region of turbulent boundary layers, and the relation of the bursts to surface pressure fluctuations and the large eddies in the outer intermittent region. Many current ideas about bursts are incompatible with the classical arguments about inner layer autonomy leading to the widely-used logarithmic laws for velocity profile and skin friction, and the practically-oriented work on pressure fluctuations done in the 1960's terminated before the development of modern conditional-sampling techniques for identifying turbulent eddies. Therefore, a careful study of inner layer behavior should have considerable scientific and practical value at the present time. In particular it should contribute to: (1) knowledge of the way in which surface pressure fluctuations are generated (with possible application to methods of drag reduction by the use of flexible surfaces; (2) belief - or disbelief - in the logarithmic laws that are so widely used for predicting "scale effect", the variation of skin



friction with Reynolds number; (3) knowledge of the generation of Reynolds stress, and hence ability to predict it. A three-year program is proposed. The study will interact with work on conditional sampling already in progress with support from the Science Research Council. This latter study has the object of measuring the generation terms in the Poisson equation for the pressure fluctuations within the turbulence (pressure-fluctuation measurements within the turbulence being of uncertain accuracy): the application is to the modeling of the "pressure strain" correlation in the Reynolds shear-stress transport equation. Both studies will use the conditional-sampling techniques already extensively developed, including the use of heat addition to mark selected parts of the turbulent fluid.

### 3033 PRESSURE ON A SLENDER, AXISYMMETRIC BODY AT HIGH ANGLES OF ATTACK IN A VERY LOW TURBULENCE AIRSTREAM

P. C. Dexter  
J. W. Flower  
University of Bristol  
SL: MICOM

Tests with a slender axisymmetric body at high incidence have shown that the use of a very low turbulence airstream can eliminate the 'switching' pattern previously noted. It was, however, found that the pressures were still dependent on roll angle, and hence that roll angle would remain an important variable throughout the investigation. Further investigations are being made using a model with many more pressure holes, and with the facility to roll the model without shutting down the tunnel. In order to make the best use of the model, and the most efficient use of the Royal Aircraft Establishment's wind tunnel, it is desirable to extend the investigation and improve the speed of data acquisition.

### 3034 NONLINEAR BURNING STABILITY OF SOLID PROPELLANTS

I. De Luca  
Politecnico di Milano  
SL: BRL SC: MICOM, ARRADCOM

This proposal is a continuation of work already performed with the European Research Office

sponsorship in the period October 1977 to September 1980. The previous activity produced 12 papers or technical notes presented at various Italian, European, USA, USSR scientific meetings. It is felt that large progress was made and further efforts are worthwhile. The ultimate purpose of this work is to formulate basic laws governing the nonlinear dynamics of solid propellant burning. This implies that: (1) findings should be readily extended to other cases of heterogeneous combustion; in particular, results are not dependent on the nature of solid propellant. (2) new flame models are not being proposed, rather a method is offered to judge how good any unsteady flame model is. (3) information regarding steady state solutions is found as a particular case. (4) interactions combustion/fluidodynamics (e. g., in a rocket combustion chamber) are not considered. It should be observed that no attempt is made to explain or predict the steady flame structure of burning propellants. This is supposed to be assigned in terms of burning rate vs pressure (experimental data) and flame temperature vs pressure (thermochemical equilibrium computations). This information, in addition to a proper flame model, is enough to define fundamental nonlinear properties of given propellants. By making the usual set of assumptions it is necessary to study the stability properties of the nonlinear heat conduction equation in the condensed phase (the standard Fourier partial differential equation with time varying boundary conditions). This is done by an approximate analytical technique, but keeping the nonlinearity of the problem. It was shown that a nonlinear algebraic function, called static restoring function, can be determined defining nonlinear static and dynamic burning stability boundaries for finite size disturbances. The dynamic stability boundaries are, in principle, restricted to the common case of forcing functions monotonically decreasing in time, but are valid for any kind of transient.

### 3037 TRANSONIC NOISE GENERATION BY DUCT AND PROFILE FLOW

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Göttingen  
SL: AMES SC: BRL, AMES

A study of the noise generation mechanisms of transonic flows in general and flows in curved ducts

and over profiles in particular will consist of high speed interferometric framing of the two-dimensional flow fields and piezo-electric transducers for pressure measurements on duct side walls. As a minimum, this approach will yield position and form of the waves in the oscillating flows in phase planes which give information on the source area of the oscillations, on feed-back loops and on wave propagation in the flow field. These experimental results will provide the basis for analytically simulating oscillating transonic systems and thus will shed light on conditions which determine the appearance of flow oscillations and thus noise and vibration in transonic flow and therefore this research represents a unique approach in flow induced vibrations and noise. The proposed research is relevant to problems involving unsteady transonic shock-boundary layer interaction and flow separation. These flow phenomena are of research interest for gas turbine compressor inlet flow and flow over helicopter blades. These flow phenomena, in a steady flow environment, are also of interest in transonic flow over conventional and hollow projectiles.

#### 3049 EVALUATION OF SERVICE LUBRICANTS AND METALS

A. Cameron  
Imperial College of Science and Technology  
SL: MERADCOM SC: AVRADCOM

Engine testing of lubricants is an expensive and time consuming process but so far the only way to obtain reliable results. The Lubrication Laboratory at Imperial College has developed completely new testing machinery designed to replace engine testing and to do much of the work in the laboratory. Three areas will be studied with these new devices: (1) Piston ring lubrication; (2) Surface fatigue under the combined stresses of tribological contact and cyclic loading; (3) Comparison of engine tested oils with new machinery. Reduction in multigrade viscosity employs an extension of the optical techniques pioneered by the Lubrication Laboratory. The work on surface fatigue is most important as it offers the possibility of shortening the time normally taken to determine fatigue life under lubricated Hertzian conditions by applying an over-riding tensile stress. The new machine which will be used has been manufactured and is now being commissioned.

#### 4006 EFFECT OF CONTROL FINS AND ANGLE OF ATTACK ON PLUME-AFTERBODY FLOW SIMULATION

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SL: MICOM SC: MICOM

Propulsive jet-external slipstream interactions or interference with solid surfaces such as occur during launch conditions have elicited research attention from the earliest experiments. In particular, the strong relationship between such interactions and near wake pressure and temperature, induced external flowfield separation, wake flowfield at angle of attack, rearward located control fin effectiveness, and launch equipment performance can give rise to undesirable aerodynamic performance. In extreme cases, plume induced separation may result in catastrophic pitch up of missiles because of loss of stability or degradation of control effectiveness. The present study is a continuation of a previous ERO Grant concerning the modeling laws for rocket or jet exhaust. The basic validity of a theoretical modeling methodology by Korst has been confirmed under restricted conditions by ongoing experimental studies. It is proposed to make use of the special characteristics of the FFA hot Freon facility to examine the limits of modeling methodologies for conditions which are beyond available computational methods yet are needed in performance prediction for full scale flight vehicles. This will include extension of current work for simple configurations in terms of the Mach number regime, angle of attack and modifications for some complex models to examine interference effects of aft mounted control surfaces.

#### D. MATERIALS

##### 2283 HIGH FREQUENCY FATIGUE BEHAVIOR OF SELECTED MATERIALS

R. Stickler  
University of Vienna  
SL: AMMRC SC: CERL, BWL

The major effort of this contract is directed towards the exploration of the high-frequency fatigue behavior of several candidate materials over the

temperature range between room temperature and temperatures in excess of 1100 C. Materials being investigated include steels, fiber reinforced metal matrix composites, and high performance structural ceramics. An investigation has begun on the critical stages of fatigue damage accumulation leading to crack initiation as a function of material, environment, temperature and loading conditions. A combined SEM and high-frequency (ultrasonic) test rig is being used in conjunction with traditional fatigue testing and metallographic techniques.

#### 2502 THE USE OF ION IMPLANTATION TO IMPROVE THE CORROSION RESISTANCE OF MAGNESIUM

V. Ashworth  
UMIST

SL: AMMRC, ONR SC: ARRADCOM, MERADCOM

Potentiostatic current-time measurements on unimplanted Mg and Ta<sup>+</sup> implanted Mg specimens at pH values of 12, 11 and 10 have been carried out. SEM studies of the specimens used have also been made. The experimental results clearly confirm the previous findings; specifically, implanted Ta is unable to form a protective Ta<sub>2</sub>O<sub>5</sub> film and instead serves to disrupt the continuity of the hydrated MgO film. Thus, current densities recorded in the potentiostatic current-time experiments are always higher for the implanted than for the unimplanted material, the differences being more marked at pH 12 and 10 than at pH 11. On the other hand, SEM studies show that both unimplanted and Ta<sup>+</sup>-implanted Mg undergo a localized type of corrosive attack which undermines the metal surface and promotes formation of small particles of metal; these become detached and eventually fall from the specimens. In summary, Ta implantation has a quantitative but not a qualitative effect on the corrosion behavior of Mg.

#### 2549 THE EFFECT OF HIGH PRESSURE ON PHASE RELATIONSHIPS AND SOME PROPERTIES OF Ti AND ITS ALLOYS

A. Rabinkin  
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SL: BWL SC: AMMRC, BRI

Changes in crystal structure induced by the application of high pressure up to 9.2 GPa (92 kbar)

at 300 K were studied in metastable Ti-Mo and Ti-V alloys in wide concentration range by transmission electron microscopy and were correlated with the results of quantitative X-ray diffraction analysis. The high pressure treatment causes formation structures. The amount of  $\omega$  phase that remains after the pressure is released is significantly larger than that which is obtained after quenching. As a result of that substantial changes in mechanical properties of the alloys takes place. An anomalous increase of the critical temperature of superconductivity  $T_c$  was observed in Ti-Mo alloys under pressure. An attempt is made to correlate the effect of alloying and pressurizing on phase stability with changes in basic physical properties of the phases.

#### 2571 ENVIRONMENTAL FAILURE OF ADHESIVE BONDING IN RESIN GLASS COMPOSITES

E. H. Andrews  
Queen Mary College  
SL: AMMRC, NLABS SC: AMES, ARRADCOM, MICOM, CERL

The adaptation of the Andrews-Stevenson (A-S) adhesion test, previously used for epoxy to metal adhesive systems, for epoxy-to-glass bonds has been achieved for epoxy resins having relatively low  $T_g$ 's (e. g. Epikote 828,  $T_g = 71^\circ\text{C}$ ). Partial success has been achieved for high  $T_g$  resins (e. g. SP250). This adaptation has required considerable sophistication of test-piece geometry to prevent thermal contraction stresses shattering the glass substrates during specimen preparation. The effect of water immersion at  $80^\circ\text{C}$  for various periods of time has been studied for the system Epikote 828 pyrex glass in the absence of coupling agents. The fracture mode becomes increasingly cohesive as immersion time increases and the peak in the curve of fracture energy versus crack speed falls progressively. Using Andrews' generalized theory, the adhesive fracture energy  $\theta$  can be separated into its interfacial energy and dissipative components. The interfacial energy  $\theta_0$  is found to decrease exponentially with time, from its cohesive value of approximately  $2.6 \text{ J/m}^2$  to some  $0.13 \text{ J/m}^2$  after 100 h immersion at  $80^\circ\text{C}$ . The nature of the decrease is indicative of a first order chemical hydrolysis reaction at the interface. The value of  $\theta_0$  after 100 h is much less than the residual value ( $\sim 0.7 \text{ J/m}^2$ ) for similar treatments when the

substrate is titanium metal rather than glass. It suggests that spontaneous separation of resin from glass may eventually occur.

## 2615 DURABILITY OF STRUCTURAL ADHESIVELY BONDED SYSTEMS

O. Ishai  
Technion Research & Development  
Foundation Ltd  
SL: AMMRC SC: MERADCOM

(a) An extended modified effective stress-strain concept has been developed, which takes into account the effects, throughout the linear, non-linear and plastic failure levels, of both the deviatoric and the volumetric stress components on the mechanical behavior of an adhesive material. This approach, which relates the tensorial components of stress and strain at each element to their respective effective values, promises to lead to a successful stress analysis and structural design of bonded joints. It permits the numerical study of the behavior of the interlaminar adhesive layer (IAL) in a state of multiaxial stress with the aid of the empirical data obtained from simple uniaxial loading. By an experimental investigation, which was conducted on bulk and in-situ structural adhesive specimens, the hypothesis was verified in certain cases. (b) The main problems involved in the adhesive bonding of structural systems stem from the low stiffness and strength of the interlaminar adhesive layer (IAL), its non-linear time-dependent mechanical behavior, and its sensitivity to temperature and humidity. Another aspect of the current study, focuses on the time-dependency of the states of stress and strain within an adhesive layer under mechanical loading up to the non-linear range. The prediction of the interlaminar stresses and strains was obtained by an interactive numerical procedure, using the finite-element method. The solution is based on a non-linear time-dependent effective loading function derived from an empirical stress-strain relationship of the adhesive material and from a postulated function describing the relationship between the stress and the strain tensors of each element, on the one hand, and their respective effective values. The findings indicate the expected trend of stress decrease and strain increase with time.

## 2618 SURFACE ACOUSTIC WAVES AND ACOUSTIC MICROSCOPY FOR N.D.T.

E. A. Ash  
University College, London  
SL: AMMRC SC: AVRADCOM, HDL, AMMRC

A second Progress Report has been released covering the period from 1st October, 1980 to 31st March, 1981. This period has been devoted to the utilization of the high frequency microscope on a number of NDE problems, and to effecting instrumental improvements, the need for which became apparent. There is no question but that for observation of defects in metals at substantial depths, (more than a few tens of microns), it is essential to work at a lower frequency. Moreover, the fact that one is then attempting to image deep inside a high velocity material, imposes special requirements on the lens design. It was decided to work at 150 MHz for problems of this nature. The lens has been designed and constructed. At this frequency there is, however, a significant difficulty arising from the need to bond transducers - since the thickness of deposited ZnO transducers would be excessive. On the other hand, 150 MHz transducers are exceedingly thin and fragile. It is, therefore, necessary to use a sophisticated bonding technique. An effective In bond has been developed. The theoretical work is continuing on two topics. The first relates to gaining a better understanding of focussing under high loss conditions. Two new avenues are being explored. Also an analysis of a phenomenon of fringe formation, which is believed to have ultimate substantial practical value, has been started.

## 2622 CHARACTERIZATION OF URANIUM-0.75% TITANIUM ALLOYS EXTRUDED FROM THE GAMMA PHASE

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SL: AMMRC SC: WA, ARRADCOM

High density and tensile properties can be achieved in U-0.75% Ti by water quenching from the  $\gamma$  phase and subsequently ageing at lower temperature. These high quench rates have been found to give rise to centerline cracking and void formation in medium and large diameter bars (i. e.



greater than approximately 20mm diameter). In the present work the effects of quench rate, mode of quenching and subsequent ageing treatments on microstructure and properties of a U-O.75% Ti alloy have been investigated. Particular emphasis has been placed upon heat treatment of 35mm diameter,  $\gamma$ -extruded, bar. Cooling rates in the range 2 - 480 Ks<sup>-1</sup> were obtained, resulting in a wide spectrum of microstructures ranging from granular  $\alpha$  after slow rates of cooling to almost fully martensitic  $\alpha'$  after faster cooling. By optimising the rate and mode of quenching the problems of centerline cracking and void formation have been circumvented. Even at the fastest cooling rates only approximately half to three quarters of the available titanium were retained in solution. Nevertheless, subsequent ageing at 450°C produced hardnesses in excess of 480 Hv<sub>30</sub> due to precipitation of U<sub>2</sub>Ti. Extrusion in the  $\gamma$  phase resulted in a pronounced texture with (002) perpendicular to the extrusion direction. This preferred orientation could be eliminated or diminished by reheating into the  $\gamma$  phase. Pronounced texturing was not produced in bars by controlled immersion. However, other work on  $\alpha$ -extruded bars has shown that it is possible to induce prominent textures by this method.

#### 2625 CRYSTALLINE DEFECTS AND DEFORMATION MECHANISMS IN SEMI-CRYSTALLINE POLYMERS

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Brunel University  
SL: AMMRC SC: CERL, MERADCOM

The main objective of the work carried out to date was to establish a sound experimental base for the proposed research, particularly with respect to thin high density polyethylene (HDPE) film preparation\*, the deformation of thin films and subsequent recording of electron diffraction patterns and electron images. This objective was successfully realized. The techniques which have to be used for preparing HDPE films for deformation studies have been satisfactorily accomplished by the investigators, and so have the electron diffraction and imaging techniques. Results are now being generated quickly, and a full and successful interpretation of the data is anticipated. All of the electron optical analysis was carried out

with a specially modified JEOL 120CC transmission electron microscope with scanning transmission attachment, in order to minimize electron beam damage of the thin spherulitic polymer films. \*Crystallization conditions are known to have a marked effect on the microstructure of polyethylene spherulites. Previous studies have investigated the deformation mechanisms operative in slowly cooled thin films whereas in the present programme the objective is to investigate the deformation of rapidly cooled polyethylene spherulites which have a characteristic banded morphology.

#### 2682 FRACTURE OF THERMOSETTING RESINS AFTER EXPOSURE TO WATER

G. Pritchard  
Kingston Polytechnic  
SL: AMMRC SC: CERL

Advanced composite materials should be able to withstand hot, wet conditions. This investigation is concerned with organic, cross linked resins and specifically with unsaturated polyester resins. The aim was to determine the extent of loss of fracture toughness of the unreinforced resins as a result of immersion in hot water. Data have been obtained for one polyester resin, immersed in water at 30°C and at 65°C for nearly two years. A theory of degradation has been formulated. The findings are currently being extended to a series of resins differing only in molecular weight distribution and in number-average molecular weight. The outcome should be the establishment of optimum molecular weight distributions.

#### 2705 IMPURITY AGGREGATION PROCESSES AND MECHANICAL BEHAVIOR IN SOLID DIELECTRICS

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University of Madrid  
SL: AMMRC SC: ETDL

The following lines of research on singly and doubly doped alkali halide crystals are underway: a) Optical spectroscopy (absorption and luminescence) as tool to investigate aggregation and precipitation processes of divalent impurities, as well as the nature of the precipitates formed. b)

Yield stress testing to follow the same processes and consequently ascertain the role of the various aggregates and precipitates on mechanical strength. c) Ionic thermocurrents (ITC) to understand the dielectric relaxation mechanisms associated with impurity complexes and precipitates. Moreover, the kinetics of dipole decay is also being studied during appropriate annealing treatments.

#### 2711 EFFECT OF CONTAMINANT ATOMS ON THE STRUCTURE OF VAPOUR-GROWN EPITAXIAL FILMS

J. P. Jones  
University College of North Wales  
SL: ETDL SC: AMMRC

Whiskers of silicon, gallium arsenide and indium phosphide are being prepared by the VLS technique to use them to form field emitter tips. Such tips are known to present unambiguously clear (100) and (111) surfaces after field evaporation of overlying impurity. Examination of these surfaces by field emission microscopy and the study of changes in their electronic characteristics produced by condensing into their surface thin layers of technologically significant metals, e. g., gold on silicon, aluminum on gallium arsenide and silver on indium phosphide are underway. The metal semiconductor junctions so formed approximate most closely existing models of the interface and so give insight into the important parameters which control the junction characteristics. An hemispherical dispersion spectrometer is being used to study the effect of thin metal layers on the density of surface states with the object of identifying interfacial states and the conditions under which they form. Parallel experiments using the Kelvin Probe on macroscopic slice material are also being done. An obviously important extension of this study to the effects of introducing small sub-monolayers amounts of impurity, e. g., oxygen will be initiated.

#### 2723 ADHERENCE AND BONDING OF THE ION PLATED FILMS

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M. G. El-Sherbiny  
University of Cairo  
SL: CERL SC: BWL, AMMRC

A detailed study is being made on nucleation, growth, interface formation and microstructure of ion plated films. Extensive graded interfaces having intermetallic compounds of constant chemical composition are observed for the ion plated coating. The mechanism of formation of these interfaces is dominated by the generation of mobile surface vacancies and the atomistic mixing process of both the evaporated film atoms and sputtered substrate atoms. A number of test specimens are being used to investigate the formation of such interfaces under different processing conditions with different metal pairs. Low energy deposition (10 $\mu$ m Hg argon & 5 Kv) as well as high energy deposition (50 $\mu$ m Hg argon & 5 Kv) are considered. Furthermore, compatible metal pairs of high solid solubility such as Cu-In and Cu-Ag as well as incompatible metal pairs of low solid solubility (less than 0.1%) such as Ag-Ni and Ag-Fe are carefully investigated. In each case, the coated test specimens are sectioned for microscopic X-ray diffraction and microprobe examinations. Further studies on incompatible metal pairs are still in progress. The optimum processing conditions leading to the formation of extensive graded interfaces have been obtained. These results indicate that high energy deposition is recommended providing that the interfacial contaminations can be avoided. Film columnar growth, pin holes, porosity, nodules and aggregates are also being investigated in an attempt to obtain the optimum processing conditions for ideal structures. Scanning electron microscopy, optical examinations and X-ray analysis of grain size are frequently used. Electron micrographs are also used in studying the surface structure. On the other hand, accelerated galvanic tests are made to measure porosity density. Finally optimum processing conditions are obtained by using a figure of merit for the characteristics of ion plated films. Test results revealed that optimum film characteristics are attainable by increasing the bias voltage and reducing both the gas pressure and deposition rate.

**2751 INFLUENCE OF IMPURITIES IN TUNGSTEN AND MATRIX COMPOSITION ON THE TUNGSTEN-MATRIX INTERFACIAL PROPERTIES OF HEAVYMETAL ALLOYS**

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W. Huppmann  
University of Vienna  
SL: AMMRC SC: ARRADCOM, BRL

The principle aim of this project is the investigation of the influence of interstitials and other impurities on the mechanical properties of heavy metals with a tungsten content higher than 95%. Since during liquid phase sintering of W heavy metals the W grains show a considerable growth, changes in their chemical composition are probable. In particular the solution-precipitation mechanisms observed may remove or concentrate impurities within the W grains. Impurities removed from the W will be enriched in the binder or, which is probably much more detrimental, at the W/binder interface where they may strongly affect the wetting between binder and grains. The sintering behavior, the resulting microstructure and the mechanical properties of the W heavy metal might be considerably influenced by such effects. The investigation carried out to date characterized the starting materials and enabled the selection of the composition for future experiments. The chemical analysis showed the similarity of the powders, except of the doping additives. SEM investigations showed slight differences especially in the grain shape and in the trend to form agglomerates. The compactibility tests confirmed the opinion expressed in the literature that larger grain sizes and broad grain size distributions give higher green densities. This is due to the very high packing density achieved by broad grain size distributions. The different compaction properties of some powders which showed pressing faults at all pressures cannot immediately be explained. The sintering tests concentrated on investigations into the optimum W content; they were carried out with samples of the standard powder W507/79 and showed that samples with almost 100% density can be achieved up to a W content of 97%; more W in the heavy metals causes high porosity and a drastic decrease in the mechanical properties. 96%W seems to offer the best compromise; the elongation is about 10% and the tensile strength is also very high, appr. 1000 MPa. The influence of the sintering temperature,

especially on density and hardness, is slight for specimens containing 96%W, but strong for high W content of 98 and 99%. The subsequent investigations will be carried out with samples containing 96%W.

**2762 BALLISTIC IMPACT RESISTANCE MULTI-LAYER TEXTILE FABRICS**

J. W. Hearle  
C. M. Leech  
University of Manchester  
SL: NARADCOM SC: AMMRC, CERL

Ballistic tests have been performed on a heavy duty nylon fabric. For each test the impact velocity was established; and if the fabric was penetrated, the energy loss of the projectile was measured. The results were compared with those from similar tests on the following: i) the same heavy duty nylon treated with either a low or a high friction finish. ii) a triaxially woven nylon fabric. iii) a lightweight parachute fabric. It is understood that Kevlar fabric is ballistically superior to nylon; however the fabric finishes were found to have a far greater effect on the yarn to yarn friction of the nylon fabric as opposed to the Kevlar. Therefore, it was decided that the initial tests should be performed on the nylon fabric. At the time of testing the triaxial fabric was only available in nylon. The following were determined:- i) the nylon fabric treated with a high friction finish was found to be ballistically superior to the same fabric treated with a low friction finish. ii) comparisons between a triaxial and a biaxial fabric showed that they have similar ballistic resistances. iii) a lightweight fabric was found to be ballistically inferior, weight for weight, than a heavy duty nylon material.

**2781 THE EFFECT OF SIMULATED WELDING CONDITIONS ON AUSTENITE DECOMPOSITION KINETICS**

F. A. Calvo  
University of Madrid  
SL: CERL SC: EOARD, CERL, AMMRC

The polished surface technique (PST) weld simulator has been used to obtain quantitative

information on the transformation taking place in the weld heat affected zone (HAZ) in Fe-0.6C material. Quantitative optical metallography has been used to determine the fraction of ferrite and pearlite in the weld HAZ as a function of distance from the center of the fusion zone. Microthermocouples spot welded to the specimen have been used to measure the thermal conditions during the welding cycle as a function of distance from the center of the fusion zone. Microstructural and thermal data are in the process of being correlated to provide a semi-quantitative description of the transformations taking place in the HAZ.

#### 2786 THE EXTENSION OF FRACTION MECHANISM MAPS TO HIGH STRAIN RATES AND THE DEVELOPMENT OF FRACTURE TOUGHNESS MAPS

M. F. Ashby  
University of Cambridge  
SL: AMMRC SC: CERL, BWL, MICOM,  
ARRADCOM

Mechanisms of deformation and of fracture at high rates of strain have been considered and the collection of pertinent data has been started. In particular an analysis was undertaken of the conditions under which adiabatic shear becomes an important mode of deformation. How this affects fracture behavior is being examined. In doing so it was found that a new kind of deformation and fracture map may be more suitable as a means of displaying and analysing these high strain rate phenomena. The axes are strain rate vs. temperature (instead of stress vs temperature), which has the effect of expanding the part of the map which contains information about high strain-rate deformation and fracture. It is anticipated that one will be able to include in such diagrams data, and theoretical ideas, about the onset of adiabatic shear. It is also expected that such maps should prove useful in analysing such problems as the forming of metals at high rates of strain; and the behavior of materials when impacted by projectiles.

#### 2820 LOW TEMPERATURE STRUCTURE AND PROPERTIES OF GRAPHITE LAMELLAR COMPOUNDS

A. Herold  
University of Nancy  
SL: MERADCOM SC: ETDL, AMMRC, CERL

The initial stage of this contract has been concerned with the setting up of a system for studying the structure of intercalation compounds down to the temperature of liquid helium. The various designs of the sophisticated X-ray and cryostat facility have been completed. The equipment has been assembled and installed. The final adjustments and test, indispensable for the precision of the measurements desired, are being completed. It is expected that the study of structure of various intercalation compounds will be started in the near future.

#### 2821 ELEVATED TEMPERATURE AQUEOUS STRESS CORROSION OF Al-Zn-Mg

H. Flower  
Imperial College  
SL: CERL, ARRADCOM SC: AMMRC, NLABS

This study will determine whether the hydrogen induced embrittlement reaction observed when Al-Zn-Mg alloys are exposed to water vapor at elevated temperatures is also responsible for the stress corrosion failure of the material in aqueous environments. Conventional stress corrosion testing and fractography will be employed to characterise properties as a function of heat treatment and corrosion conditions. However, the main emphasis of the work is microstructural and will require the use of the high voltage electron microscope (HVEM) to examine the microstructure of the crack tip in foils prepared from the bulk and to carry out in situ stress corrosion testing using the environmental cell and a straining stage. The chemistry of the crack path, corrosion products and hydrogen trap sites will be determined using an analytical scanning transmission electron microscope (TEMSCAN). This contract has had its start delayed until October 1981.



**2840 DEVELOPMENT OF HIGH-MODULUS  
POLYDIACETYLENE FIBRES FOR USE  
IN FIBRE-REINFORCED  
COMPOSITES**

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Queen Mary College  
SL: AMMRC SC: NLABS, MERADCOM,  
ARRADCOM

It is possible to make high-modulus polydiacetylene fibres by the solid-state polymerization of certain substituted diacetylene monomers. A detailed programme has been started in which new polymer fibres will be synthesised and their mechanical properties evaluated. Composites incorporating the fibres will be fabricated and mechanical performance will be studied. The fibres produced so far are in the form of short strands, several centimeters in length. Techniques of producing continuous fibres will be investigated.

**2880 FACTORS CONTROLLING WELD  
METAL CHEMISTRY**

N. Christensen  
University of Trondheim  
SL: CERL SC: ARRADCOM, AMMRC, CERL

This project is concerned with equilibrium aspects of the reactions controlling the weld metal composition. Information on the approach to pseudo-equilibrium in welding processes, or in certain cases possibly the lack of such approach would assist in a rational design of consumables to attain a desired composition. It is intended to test experimentally a simple model for predicting, or at least estimating the extent of chemical interaction of liquid weld metal with its surroundings (slag or arc atmosphere). This model is based on the assumption of close approach to local chemical equilibrium during the high-temperature stage of the welding process and readjustment by precipitation in the subsequent stage of cooling.

**3005 GLASS FIBRE/EPOXY RESIN  
INTERFACE LIFE-TIME PREDICTION**

K. H. G. Ashbee  
University of Bristol  
SL: AMMRC SC: CERL

Using optical retardation measurements in order to monitor axial stress in individual fibres, it has been

possible to make direct comparisons of loss of interfacial load transfer during water uptake. These data are now being used as a basis for investigating the possibility of interfacial lifetime prediction, including life-time prediction during natural weathering. An attempt will also be made to include the accelerating effects of externally applied stress. Specifically, the nature and magnitude of mechanical damping local to the interface will be investigated in order to determine the physical mechanisms, and laws which describe accumulation of interfacial damage during the cyclic stressing of composites in humid atmospheres.

**3021 SILICON OXYNITRIDE GLASSES AND  
GLASS-CERAMICS - PREPARATION  
AND PROPERTIES**

R. Abramovici  
Israel Ceramic and Silicate Institute  
SL: AMMRC SC: CERL

Incorporating of nitrogen in glasses of various silicate systems leads to increased viscosity, density, annealing point, indentation hardness, electrical resistivity and to lower thermal expansion coefficient and dielectric losses. These changes of the properties are attributed to the fact that nitrogen is chemically bonded to silicon in the glass network. Replacement of oxygen by nitrogen causes a more highly cross-linked structure. Beside phases specific for devitrification of glasses without nitrogen, new phases, some of them unidentified, were found in the corresponding crystallized oxynitride glasses. The aim of the present work was to study the influence of increasing amounts of nitrogen (added to Si-N) on some glass properties, before and after heat treatment for crystallization. It was started with a glass melting at low temperature with composition corresponding to the low temperature eutectic (975°C) in the SiO<sub>2</sub>-Al<sub>2</sub>O<sub>3</sub>-Li<sub>2</sub>O system. The base glass was compared to glasses containing 2.44 and 4.74 at % N. Taking into account the crystallization capacity of glasses with high lithium content, on one hand, and the hints from the literature concerning the self nucleating capacity of oxynitride glasses on the other hand, glasses without nucleating agent were studied in the first stage of this work. Preliminary results indicate that addition of nitrogen to the

studied glasses, even in small ratios, induces an increase in viscosity of the melt, which agrees with published data. At the same time, the thermal expansion decreases, less for untreated, more for crystallized glasses. The presence of nitrogen shifts structural changes in the studied glasses towards

higher temperatures. The qualitative phase composition is roughly similar for all the three crystallized glasses. It seems that the presence of nitrogen enhances more intensive crystallization of eucryptite. Up to 4.74 at % N, this remains incorporated into the glass and not crystallized out.

# Proposal Numbers

*Note:* The Roman numeral after each proposal number refers to the scientific division; the alphabetic character following

the Roman numeral refers to the division subsection. Proposal numbers are in ascending sequence within the subsections.

| Proposal No. |       |          |       |          |       |          |       |
|--------------|-------|----------|-------|----------|-------|----------|-------|
| 02283        | V-D   | 14889-MS | IV-B  | 15979-MS | IV-C  | 16719-E  | III-B |
| 02376        | V-A   | 14925-MS | IV-A  | 15989-E  | III-A | 16728-C  | I-E   |
| 02502        | V-D   | 14926-L  | II-A  | 15992-L  | II-C  | 16729-L  | II-E  |
| 02522        | V-A   | 14932-E  | III-A | 15999-MS | IV-D  | 16740-MS | IV-D  |
| 02526        | V-C   | 14990-E  | III-B | 16002-MS | IV-A  | 16742-C  | I-B   |
| 02549        | V-D   | 15048-E  | III-C | 16004-C  | I-B   | 16746-C  | I-E   |
| 02571        | V-D   | 15078-E  | III-A | 16005-E  | III-A | 16760-E  | III-B |
| 02600        | V-C   | 15098-MS | IV-B  | 16017-C  | I-D   | 16765-L  | II-A  |
| 02615        | V-D   | 15104-MS | IV-C  | 16030-E  | III-B | 16780-C  | I-D   |
| 02618        | V-D   | 15112-MS | IV-B  | 16036-E  | III-A | 16782-MS | IV-C  |
| 02622        | V-D   | 15124-MS | IV-D  | 16058-MS | IV-D  | 16791-E  | III-C |
| 02625        | V-D   | 15226-E  | III-A | 16061-E  | III-B | 16796-C  | I-E   |
| 02639        | V-C   | 15243-E  | III-A | 16062-MS | IV-C  | 16803-E  | III-A |
| 02646        | V-C   | 15299-MS | IV-B  | 16086-C  | I-E   | 16810-MS | IV-B  |
| 02652        | V-D   | 15319-L  | II-F  | 16089-MS | IV-D  | 16822-MS | IV-D  |
| 02687        | V-A   | 15336-E  | III-B | 16091-E  | III-A | 16824-MS | IV-C  |
| 02689        | V-C   | 15400-E  | III-A | 16101-E  | III-B | 16841-E  | III-B |
| 02690        | V-C   | 15403-E  | III-C | 16110-C  | I-D   | 16850-MS | IV-B  |
| 02693        | V-A   | 15427-E  | III-A | 16113-L  | II-C  | 16856-C  | I-D   |
| 02696        | V-A   | 15433-MS | IV-D  | 16116-C  | I-A   | 16862-E  | III-A |
| 02705        | V-D   | 15464-L  | II-D  | 16123-MS | IV-B  | 16865-C  | I-A   |
| 02711        | V-D   | 15466-C  | I-E   | 16124-C  | I-D   | 16867-L  | II-E  |
| 02723        | V-D   | 15489-MS | IV-A  | 16133-C  | I-B   | 16881-MS | IV-D  |
| 02736        | V-C   | 15513-E  | III-A | 16143-MS | IV-B  | 16885-MS | IV-D  |
| 02741        | V-A   | 15521-C  | I-C   | 16144-C  | I-E   | 16911-MS | IV-D  |
| 02751        | V-D   | 15525-L  | II-C  | 16146-L  | II-D  | 16914-E  | III-C |
| 02762        | V-D   | 15532-E  | III-C | 16184-C  | I-B   | 16916-MS | IV-C  |
| 02781        | V-D   | 15539-MS | IV-A  | 16191-C  | I-A   | 16930-C  | I-D   |
| 02786        | V-D   | 15557-C  | I-D   | 16192-C  | I-A   | 16931-C  | I-C   |
| 02792        | V-A   | 15591-C  | I-D   | 16196-C  | I-B   | 16957-E  | III-C |
| 02794        | V-A   | 15601-C  | I-E   | 16201-C  | I-B   | 16961-C  | I-E   |
| 02798        | V-A   | 15611-C  | I-A   | 16227-MS | IV-B  | 16964-MS | IV-E  |
| 02813        | V-A   | 15618-C  | I-B   | 16232-C  | I-E   | 16972-E  | III-B |
| 02817        | V-A   | 15628-MS | IV-B  | 16254-E  | III-A | 16983-E  | III-C |
| 02820        | V-D   | 15643-MS | IV-B  | 16256-C  | I-A   | 17001-E  | III-C |
| 02821        | V-D   | 15657-E  | III-B | 16266-C  | I-A   | 17029-L  | II-A  |
| 02835        | V-C   | 15662-E  | III-C | 16272-L  | II-F  | 17034-E  | III-C |
| 02840        | V-D   | 15671-L  | II-A  | 16273-C  | I-C   | 17042-C  | I-A   |
| 02845        | V-A   | 15689-MS | IV-B  | 16280-E  | III-B | 17048-E  | III-B |
| 02865        | V-A   | 15690-C  | I-B   | 16294-E  | III-B | 17055-E  | III-A |
| 02872        | V-C   | 15703-C  | I-C   | 16296-MS | IV-C  | 17061-L  | II-A  |
| 02874        | V-A   | 15710-E  | III-B | 16318-C  | I-C   | 17062-E  | III-C |
| 02876        | V-A   | 15722-E  | III-A | 16348-MS | IV-C  | 17064-E  | III-A |
| 02880        | V-D   | 15743-E  | III-B | 16355-MS | IV-D  | 17067-E  | III-A |
| 02885        | V-C   | 15746-MS | IV-E  | 16357-C  | I-E   | 17069-C  | I-A   |
| 02887        | V-C   | 15761-L  | II-D  | 16365-C  | I-B   | 17080-C  | I-C   |
| 02890        | V-A   | 15770-C  | I-E   | 16382-C  | I-B   | 17082-MS | IV-A  |
| 03005        | V-D   | 15771-MS | IV-D  | 16389-C  | I-E   | 17085-L  | II-E  |
| 03012        | V-C   | 15780-E  | III-B | 16391-C  | I-B   | 17108-C  | I-A   |
| 03017        | V-C   | 15783-C  | I-A   | 16423-MS | IV-D  | 17126-E  | III-A |
| 03021        | V-D   | 15787-E  | III-A | 16425-MS | IV-B  | 17134-E  | III-B |
| 03033        | V-C   | 15788-E  | III-C | 16426-C  | I-E   | 17147-C  | I-C   |
| 03034        | V-C   | 15795-C  | I-E   | 16438-MS | IV-D  | 17154-C  | I-A   |
| 03037        | V-C   | 15804-MS | IV-D  | 16448-MS | IV-D  | 17161-MS | IV-C  |
| 03047        | V-A   | 15812-MS | IV-A  | 16527-C  | I-B   | 17165-MS | IV-D  |
| 03049        | V-C   | 15824-MS | IV-D  | 16556-MS | IV-B  | 17215-MS | IV-D  |
| 04006        | V-C   | 15826-C  | I-D   | 16577-C  | I-E   | 17216-E  | III-A |
| 13653-E      | III-C | 15866-E  | III-B | 16583-L  | II-D  | 17221-E  | III-A |
| 13935-E      | III-C | 15868-E  | III-A | 16592-L  | II-C  | 17226-E  | III-B |
| 14081-C      | I-D   | 15872-E  | III-A | 16595-E  | III-B | 17247-E  | III-C |
| 14254-E      | III-B | 15879-C  | I-D   | 16601-L  | II-C  | 17267-E  | III-C |
| 14638-MS     | IV-D  | 15893-L  | II-F  | 16631-C  | I-A   | 17283-C  | I-B   |
| 14679-MS     | IV-A  | 15904-L  | II-A  | 16648-L  | II-A  | 17298-MS | IV-D  |
| 14700-E      | III-A | 15905-E  | III-B | 16651-E  | III-B | 17312-C  | I-E   |
| 14740-MS     | IV-A  | 15911-F  | III-A | 16662-C  | I-C   | 17348-E  | III-B |
| 14741-E      | III-B | 15916-MS | IV-C  | 16665-C  | I-D   | 17349-C  | I-C   |
| 14766-MS     | IV-A  | 15917-C  | I-B   | 16666-E  | III-A | 17355-C  | I-C   |
| 14858-MS     | IV-C  | 15947-L  | II-D  | 16667-MS | IV-B  | 17357-E  | III-A |
| 14868-MS     | IV-C  | 15976-C  | I-A   | 16689-L  | II-F  | 17369-C  | I-D   |

# Indices

# Proposal Numbers

|          |       |          |       |          |       |          |       |
|----------|-------|----------|-------|----------|-------|----------|-------|
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| 17378-E  | III-C | 17579-C  | I-B   | 17780-MS | IV-D  | 18001-C  | I-E   |
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| 17398-C  | I-A   | 17623-C  | I-D   | 17830-E  | III-A | 18039-C  | I-D   |
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| 17415-MS | IV-A  | 17647-E  | III-A | 17858-C  | I-B   | 18053-MS | IV-B  |
| 17416-C  | I-D   | 17650-C  | I-A   | 17861-C  | I-C   | 18055-C  | I-A   |
| 17421-MS | IV-A  | 17653-MS | IV-B  | 17877-MS | IV-C  | 18090-C  | I-B   |
| 17425-E  | III-B | 17654-C  | I-A   | 17887-MS | IV-B  | 18126-L  | II-D  |
| 17438-C  | I-E   | 17689-E  | III-A | 17899-C  | I-B   | 18139-C  | I-D   |
| 17439-MS | IV-C  | 17690-E  | III-B | 17904-L  | II-A  | 18151-C  | I-A   |
| 17451-C  | I-C   | 17698-E  | III-B | 17905-C  | I-A   | 18172-C  | I-D   |
| 17455-MS | IV-C  | 17710-C  | I-D   | 17910-L  | II-A  | 18184-MS | IV-B  |
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| 17546-MS | IV-D  | 17752-MS | IV-B  | 17973-MS | IV-D  | 18241-L  | II-D  |
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| I-B-17582       | I-C-17451       | IV-D-16638                         | III-A-15722(SL)             |
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